AMRL-TR-69-18

IDENTIFICATION OF VOLATILE CONTAMINANTS OF SPACE CABIN MATERIALS

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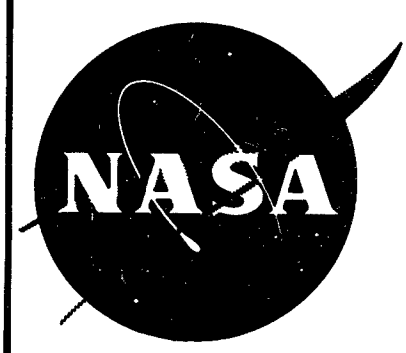
JOINT NASA/USAF STUDY



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13. ABSTRACT

Seventy-two candidate space cabin materials were tested to determine weight-loss characteristics and to identify their gas-off products. The materials were screened initially, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to 68 ± 2°C) for 24 hours in 5 psia nitrogen, to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials were then stored in 9-liter chambers at 68 ± 2°C for 72 hours and at 25 ± 2°C for periods of 30 and 60 days. Atmosphere in the chambers was oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials were identified by combinations of gas chromatography and mass spectrometry.

An additional study was made to determine the decomposition temperatures and products of degradation for carboxynitroso rubber.

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FOREWORD

The study was conducted at the Dayton Laboratory of Monsanto Research Corporation, Dayton, Ohio, under Contract No. F33615-67-C-1357. The principal investigator was Mr. F. Neil Hodgson for Monsanto Research Corporation. The study was started in January 1968 and was completed in January 1969. The entire study was under the project leadership of Mr. John V. Pustinger, Jr. of Monsanto Research Corporation.

This research was initiated by the Chemical Hazards Branch, Toxic Hazards Division in support of Project 6302, "Toxic Hazards of Propellants and Materials," Task 630204, "Environmental Pollution," Work Unit 008, "Identification of Volatile Contaminants from Space Cabin Materials." Dr. Gerd A. Kleineberg of the Chemical Hazards Branch was the contract monitor for the Aerospace Medical Research Laboratory.

This is the fourth of a series of reports on the identification of volatile contaminants of space cabin materials. Previous reports were AMRL-TR-66-53, AMRL-TR-67-58, and AMRL-TR-68-27.

This technical report has been reviewed and is approved.

CLYDE H. KRATOCHVIL, Colonel, USAF, MC
Commander
Aerospace Medical Research Laboratory

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SECTION I

INTRODUCTION

As a continuation of a series of material evaluation studies (refs. 1,2,3), 72 candidate space cabin materials were tested to determine weight-loss characteristics and to identify their gas-off products. The materials were screened initially, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to 68 ± 2°C) for 24 hours in 5 psia nitrogen, to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials were then stored in 9-liter chambers at 68 ± 2°C for 72 hours and at 25 ± 2°C for periods of 30 and 60 days. Atmosphere in the chambers was oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials were identified by combinations of gas chromatography and mass spectrometry.

An additional study was made to determine the decomposition temperatures and products of degradation for carboxynitroso rubber.

SECTION II

GAS-OFF EXPERIMENTS

A. EXPERIMENTAL METHOD

1. Types of Candidate Materials and Sample Preparation

Table I lists the candidate materials for cabin construction used in these experiments; all materials tested are commercial products provided by the Government. In most cases, the materials were prepared by the Air Force prior to testing. Whenever sample preparation was required, as in the case of some paints or two-part resins, mixing or curing was accomplished according to procedures provided by the manufacturers or the Air Force.

Materials such as paints and inks were applied to an aluminum foil substrate and subsequently tested.

Specimens used for thermogravimetric analysis (TGA) were conditioned at 23°C in a desiccator over phosphorus pentoxide for 24 hours prior to testing. For storage tests at 72 hours, 30 days, and 60 days, no pretreatment of samples was performed beyond the curing procedures cited by the manufacturers or the Air Force. The procedure for preconditioning the TGA specimens was devised to minimize adsorbed water and to put all samples on the same basis for comparing relative weight loss.

For storage tests of 72 hours, 30 days, and 60 days, a weighed portion of each sample was placed into a 9-liter chamber in such a manner as to expose the largest possible surface area. Generally, approximately 10-gram specimens were used; however, in cases where less sample was available, or when the bulk volume of the sample was excessively large, smaller specimens were used. When the bulk volume was too large and subdividing was necessary, freshly exposed surfaces were further cured at ambient conditions, i.e., 23°C and atmospheric air pressure, for 30 days or a minimum of 14 days.

Individual specimens of each candidate material were contained in 9-liter, borosilicate glass chambers for periods of 30 and 60 days at 25 ± 3°C, and for a period of 72 hours at 68 ± 2°C, under an oxygen atmosphere at 5 psia and 20-40% relative humidity. The chamber design and pretreatment of the chambers were the same as reported earlier (ref. 1). Control chambers (containing only aluminum foil) were processed concurrently with those chambers containing the test materials. No contamination was detected from the control chambers.

Table I

SPACE CABIN TEST MATERIALS

Code No.	Material
AF 024	Epon 828/Versamid 140
AF 129	Polyester Glass F141
AF 225	Corfil 615 Adhesive
AF 246	Kel-F (QSL)
AF 250	Vinylidene fluoride, Kaynar
AF 256	Plex 55 Acrylic
AF 381	Invelco 33F Fluorosilicone Grease
AF 407	Silicone Elastomer Q2-0078
AF 410	EPI Rex 510 Paint
AF 417	Diallyl Phthalate 52
AF 465	Huniseal 1B12 Laminate
AF 491	Tape-Temp-R-Glass
AF 496	Tape-Temp-R-Tape
AF 532	Lexan 101-01
AF 616	Locquic Primer Grade N
AF 619	A2 Epoxy Adhesive and Activator A
AF 623	1151 Flex Tubing (Silicone Rubber on Glass)
AF 631	Kel F (Carmer)
AF 639	CHR 3320 Silicone/Glass
AF 643	9711 Silicone Rubber
AF 645	DC-5 Lube
AF 646	DC-33 Light Grease
AF 647	DC-33 Medium Grease
AF 648	DC-33 Heavy Grease
AF 649	DC-510 Lube (Silicone)
AF 653	RTV 501 Potting Compound
AF 655	RTV 732 Potting Compound
AF 658	Silastic 8164
AF 669-9	Stycast 1090/Cat 9
AF 669-11	Stycast 1090/Cat 11
AF 671	Stycast 2762 Potting Compound
AF 675	Urelane 5712
AF 678	RTV-511 Silicone Rubber
AF 680	SE 550 Silicone Elastomer
AF 681	SF 565 Silicone Rubber
AF 699	Hysol 0151 Sealant
AF 711	Dry Film 350
AF 712	N3031A (Lubeco, Inc.)
AF 713	S1-350A-Lubricant
AF 718	401-F1 Yellow Velvet Paint
AF 719	401-H2 Blue Velvet Paint
AF 720	401-J1 Tan Velvet Paint
AF 722	425 Press. Sens. Tape
AF 725	EC-880 Adhesive
AF 727	Scotchcast 3
AF 728	Scotchcast 263
AF 730	Scotchweld 583
AF 733	Molycote X15 Dry Film Lube
AF 736	E18 Fiberglass Cord
AF 743	EE-6379 Tape (Polyimide Film)
AF 749	EPR Elastomer
AF 751	PRC-1201Q Sealer
AF 761	F55AP14 Epoxy Enamel
AF 764	F55WP20 Epoxy Paint
AF 766	U40-VD-5 Paint
AF 786	Sylgard 182 Potting Compound
AF 955	DC93-500 Part A & B
DAC-026	
DAC-029	
DAC-030	
DAC-031	
DAC-032	
DAC-033	
DAC-035	
DAC-036	
DAC-037	
DAC-039	
DAC-042	
DAC-044	
DAC-045	
DAC-101	
DAC-102	

2. Preparation of Chamber Atmospheres

After insertion of each specimen, the 9-liter test chamber was filled to a pressure of one atmosphere with oxygen saturated with water vapor. The gas was saturated with water by bubbling 99.5% oxygen (conforming to Type I of MIL-O-27210) through triple distilled water at 23°C. Test conditions were attained by subsequently reducing the pressure in the chamber to 5 psia, resulting in a test atmosphere of oxygen at 5 psia with a relative humidity of approximately 33%.

Measurement of relative humidity was made with an Alnor Type 7300 Dew-Pointer (Illinois Testing Laboratories, Inc., Chicago, Ill.).

Test atmospheres were maintained at 25° ± 3°C by storing chambers in a temperature-controlled room for periods of 30 and 60 days. The chambers that were tested at 68° ± 2°C were stored in a constant-temperature cabinet (Blue M Electric Co., Stabil-Therm DL132C).

3. Analytical Methods

Methods of analyses used in this program have been described elsewhere (ref. 1,2,3) and are summarized below.

a. Weight Loss Measurements

Conditional screening of candidate mateials was performed by measuring the weight loss of the material, using thermogravimetric measurements (TGA). Weight loss from approximately 10 g of a material was recorded continuously as the temperature of its environment was raised from ambient (approximately 23°C) to 68° ± 1°C in 4 hours and then maintained at 68° ± 1°C for 20 hours or until weight remained constant for 2 hours. All TGA measurements were made in dried, prepurified nitrogen at 5 psia.

Thermogravimetric measurements were made with a Cahn RH Electrobalance equipped with a modified F&M Model 240-00 Power Proportioning Temperature Programmer, Flo-Thru tube, a temperature programmed oil bath, and a 1 mv recorder (ref. 3).

Water evolving from the sample was monitored continuously with a Panametrics Hygrometer, Model 1000. The probe of the hygrometer was located at the sample site. Probe response under test conditions was calibrated against weight loss measurement for known amounts of water by using the Cahn electrobalance.

b. Gas Chromatographic Analysis of Gas-Off Products

Carbon monoxide, methane, and gas chromatographic analyses were performed by techniques reported earlier (refs. 1,2,3). All atmospheres in the test chamber were sampled for analysis at the temperature of the test, i.e., 25°C or 68°C.

The general analyses of the gas-off products by gas chromatography were performed on an F&M Model 810 Research Gas Chromatograph equipped with dual flame ionization detectors and a general purpose column, 20-ft x 0.25-in. ss., 20% Triton X-305 on 60/80 mesh Gas Chrom Z.

Quantitative gas chromatography data were obtained by comparing the peak heights with those of a standard mixture. Gas chromatographic instrument conditions are presented in Appendix III, Table LIX.

Identifications of gas chromatographic components were made by mass spectrometric analysis of the gas chromatographic effluent. In most cases, component identification was accomplished by the direct, tandem coupling of a fast scan mass spectrometer, CEC 21-104, to the gas chromatograph. By splitting the effluent, a portion was directed to the flame ionization detector and a second portion was introduced directly into the mass spectrometer. With some samples, a concentration step requiring the cryogenic trapping of the major portion of the total 9-liter volume was necessary. This condensate was subsequently separated into its components and characterized by the coupled gas chromatograph-mass spectrometer system.

c. Mass Spectrometric Analysis of Gas-Off Products

Two types of mass spectrometric analyses were performed for each sample. A composite analysis (ref. 1) of the atmosphere of each 9-liter bottle was made on an aliquot (125 cc) of the atmosphere with a Consolidated Electrodynamics Corporation Model 21-103C Mass Spectrometer. As indicated in Section II-A-3-b, a fast scan Consolidated Electrodynamics Corporation Model 21-104 Mass Spectrometer was used in a direct couple with a gas chromatograph. Both approaches are necessary to insure complete characterization of the chamber atmospheres.

Identification of individual components was made by mass spectrometry, supported by infrared absorption and gas chromatographic data as needed. Most of the mass spectra obtained were compared to the American Petroleum Institute (API) reference spectra. In cases where the required mass spectrum did not appear in the API collection, comparison was made with spectra from our laboratory files or from the literature.

B. RESULTS AND DISCUSSION

Weight loss data, obtained from thermogravimetric measurements, are reported in Table II for 64 materials. Eight additional samples, DAC-032, DAC-035, AF 410, AF 655, AF 671, AF 733, AF 751, and AF 955, were tested only in the bench scale 9-liter chambers. No weight loss data were obtained.

Materials listed in Table II as having no weight loss or a weight loss of <0.001% were excluded conditionally from further testing.

Partial test data (weight loss measurement) are reported for several materials (AF 719, AF 720, and AF 722) which were withdrawn from further testing by the Air Force on the basis of no longer being required.

Materials for which only carbon monoxide or methane were detected are listed in Table III.

Materials showing weight loss in excess of 1% and for which no analytical data are reported are listed in Table IV. Although these materials were conditionally screened and eliminated from further testing based on TGA measurements, all candidate materials in this program were processed in storage tests for 72 hours, 30 days, and 60 days, and their gas-off products were analyzed with gas chromatography and mass spectrometry to confirm the results of the TGA measurements. In most cases, where the weight loss exceeded 1%, gas chromatographic and mass spectrometric data were obtained only to establish the magnitude of the total off-gases. No specific identifications or measurement of quantities of individual components were made.

Two materials (AF 680 and AF 725) exhibiting weight loss in excess of 1% were treated in the gas-off chambers and their volatile products characterized and measured quantitatively to show the relationship between the TGA data and the analyses of the volatiles. The comparison of results, considering the differences between the test conditions, are good. In general, a comparison of weight loss data (23° to 68°C) with the quantitative analyses of volatiles from storage tests at 68°C should be made with care. Several opposing effects should be noted. A large number of materials continue to desorb considerable quantities of volatiles after 24 hours. Thus, more gas-off products should be present in the atmosphere during 72-hour tests than gas-off products detected as weight loss during 24-hour TGA measurements. However, adsorption of volatiles on chamber surfaces results in an opposing effect. Considerable quantities of polar and relatively non-volatile gas-off products are

Table II

WEIGHT LOSS DATA FOR CANDIDATE SPACE CABIN MATERIALS

(Obtained from Thermogravimetric Measurements)

Sample No.	Wt. of Sample (g)	Total Wt. Loss (mg)	Wt. Loss Due to H_2O (mg)	Wt. Loss Exclusive of H_2O (mg)	Wt. Loss Exclusive of H_2O (%)
DAC-026	9.8296	16.72	6.6	10.1	0.10
DAC-029	9.4527	23.20	12.5	10.7	0.11
DAC-030	10.1023	23.00	ND	23.0	0.22
DAC-031	9.6889	None	ND	-	-
DAC-033	10.7174	14.02	5.3	8.7	0.082
DAC-036	10.6603	6.85	6.7	0.1	0.002
DAC-037	11.1720	4.68	3.0	1.6	0.015
DAC-039	9.8190	8.08	1.5	6.6	0.067
DAC-042	10.2028	12.96	4.8	8.2	0.080
DAC-044	7.9560	5.72	5.3	0.4	0.005
DAC-045	10.1837	4.80	4.7	0.1	0.001
DAC-101	9.5120	None	ND	-	-
DAC-102	8.1055	121.92	24.4	97.5	1.20
AF 024	7.5962	15.00	13.1	1.9	0.025
AF 129	8.0533	22.84	1.8	21.0	0.26
AF 225[a]	7.8779	115.30	110.7	4.6	0.058
AF 225[b]	8.0939	107.60	101.9	5.7	0.070
AF 246	10.1556	None	ND	-	-
AF 250	10.0140	0.86	0.5	0.4	0.004
AF 256	10.0859	25.32	24.5	0.8	0.008
AF 381	3.7203	None	ND	-	-
AF 407	8.5350	11.84	11.0	0.8	0.009
AF 417	5.1508	14.70	1.0	13.7	0.26
AF 465	6.7777	23.82	1.9	21.9	0.32
AF 491	10.1321	0.56	0.2	0.4	0.004
AF 496	9.9156	None	ND	-	-
AF 532	9.9917	3.40	2.5	0.9	0.009
AF 616	0.1783	25.25	2.8	22.4	12.59
AF 619	10.2945	7.42	6.9	0.5	0.005
AF 623	10.9926	4.56	4.5	0.06	<0.001
AF 631	6.7193	None	ND	-	-
AF 639	9.6219	6.80	6.5	0.3	0.003
AF 643	8.3022	238.60	26.4	212.2	2.56
AF 645	11.6142	16.20	4.0	12.2	0.11
AF 646	9.6525	13.24	6.1	7.1	0.074
AF 647	10.0667	19.12	8.1	11.0	0.11
AF 648	9.1242	35.04	8.5	26.5	0.29
AF 649	6.9971	2.44	ND	2.4	0.035
AF 653	10.0607	19.22	ND	19.2	0.19
AF 658	10.0965	3.96	ND	4.0	0.040
AF 669-9	7.2966	28.50	27.6	0.9	0.01
AF 669-11	10.7313	17.36	16.5	0.9	0.008
AF 675	10.6037	22.00	16.4	5.6	0.052
AF 678	9.6593	9.44	2.4	7.0	0.072
AF 680	10.1077	216.50	6.7	209.8	2.08
AF 681	10.6231	40.05	15.4	24.6	0.23
AF 699	10.9150	60.88	55.0	5.9	0.054
AF 711	1.0900	47.28	2.3	45.0	4.12
AF 712	5.7526	122.25	6.3	115.9	2.01
AF 713	1.6112	39.30	8.8	30.5	1.89
AF 718	9.8587	190.60	189.1	1.5	0.015
AF 719	9.2323	152.50	33.6	118.9	1.29
AF 720	10.2294	88.62	72.8	15.8	0.15
AF 722	9.3129	3.68	2.0	1.7	0.018
AF 725	10.3658	130.28	10.1	120.2	1.16
AF 727	10.5641	3.36	1.0	2.4	0.023
AF 728	10.0717	7.82	7.6	0.2	0.002
AF 730	6.6820	23.04	7.0	16.0	0.24
AF 736	12.1023	4.96	4.8	0.2	0.001
AF 743	7.8353	6.12	5.0	1.1	0.014
AF 749	9.1003	30.96	9.4	21.6	0.24
AF 761	9.8040	128.50	35.7	92.8	0.95
AF 764	9.8569	186.00	38.0	148.0	1.50
AF 766	6.4900	175.42	23.1	152.3	2.35
AF 786	11.1972	8.84	7.5	1.3	0.012

[a]Undried specimen.

[b]Dried over P_2O_5 for 24 hours.

Table III

CANDIDATE MATERIALS WHICH YIELD ONLY
CARBON MONOXIDE AND METHANE - 72 HOURS @ 68°C

Sample No.	Carbon Monoxide (mg/10 gms)	Methane (mg/10 gms)
DAC 035	0.010	0.03
DAC 036	0.005	0.01
AF 381	0.001	0.01
AF 733	0.002	0.03
AF 736	0.002	0.03

Table IV

CANDIDATE MATERIALS CONDITIONALLY WITHDRAWN FROM FURTHER TESTING

(Based on Excessive Weight Loss and Preliminary Gas Chromatographic and Mass Spectrometric Measurements)

DAC 102	AF 712
AF 616	AF 713
AF 643	AF 764
AF 711	AF 766

retained on the chamber surfaces in the storage tests even at 68°C. Oily films were deposited on chamber walls in many of the tests. This effect was particularly noticeable when testing silicones in that relatively low molecular weight silicone oil condensed on the chamber surfaces. Phenomena of this type, i.e., adsorption of gas-off products such as silicone oils on chamber walls, may account for the relatively large weight-loss observed in the TGA measurements of DAC-030 and the low analytical results for H_2O and volatile gas-off products.

In general, most of the water evolved during the thermogravimetric measurements occurred in the first 4-6 hours. However, as noted for samples AF 225 (Figures 5 and 6), AF 718 (Figure 36), and AF 720 (Figure 39), considerable water was evolved throughout the 24-hour test period. These data indicate a continuous evolution of water, undoubtedly as part of the basic curing process.

Samples AF 225 and AF 719 were tested twice by thermogravimetric analysis,

(a) specimen as received (not dried)

(b) specimen dried for 24 hours over P_2O_5

The results are shown graphically in Figures 5, 6, 37, and 38. As expected, less water is detected in the dried specimens, but the weight loss profiles follow the general pattern for the undried specimens. Differences are attributable to adsorbed water, which was removed in drying the specimens over P_2O_5 but was evolved from the undried specimens at the lower temperatures during the TGA measurements.

Table V lists the types of compounds detected in the chamber atmospheres. These data represent compounds exclusive of H_2O, CO_2, O_2, and N_2.

During the TGA test for sample AF 680, small, colorless crystals were observed as deposits on the Flo-Thru tube walls. These crystals were collected and identified by infrared spectrophotometry, Figure 1, as benzoyl peroxide.

Analytical data are presented in Appendix I, Figures 3 to 62 (TGA and Water Loss Curves); Appendix II, Tables IX to LVIII (Analytical Results for Gas-Off Experiments); and Appendix III, Figures 63 to 112 (Gas Chromatograms for Gas-Off Experiments).

All values appearing in the tables of Appendix II are calculated on the basis of the dried or cured samples (this is important in the case of paints and coatings where the weight of the material is substantially reduced by drying).

Table V

TYPES OF COMPOUNDS DETECTED

I. Inorganics

Carbon Monoxide

II. Alkanes

Methane
Pentane
Hexane
Heptane
Dimethylpentane
Methylpentane
C_3-C_{15} Saturated Hydrocarbons

III. Alkenes

Chloroethylene
Ethylene
Dimethylpentene
Methylpentene
Methylhexene
Hexene
C_3 Unsaturated Hydrocarbons
C_4 " "
C_5 " "
C_6 " "
C_7 " "
C_8 " "
C_{9-15} " "

IV. Alcohols

n-Propanol
Methanol
Ethanol
2-Butanol
n-Butanol
2-Propanol
2-n-Butoxyethanol
t-Butanol
2-Ethoxyethanol
2-Phenyl-2-propanol
Diethoxyethanol

V. Alkyl Halides

1,1-Difluoroethane
Dichlorodifluoromethane

VI. Carboxylic Acids and Their Derivatives

Acetic Acid
2-Ethoxyethylacetate
2-(2-Ethoxyethoxy)ethylacetate
Methylacetate

VII. Aldehydes

Butyraldehyde

VIII. Ketones

Acetone
2-Butanone
Cyclohexanone
Hexanone
Methylisobutylketone
4-Methyl-2-pentanone
2,4-Pentanedione
Acetophenone

IX. Ethers

Methyl sec-Butyl Ether
2-Methyl-1,3-dioxalane

X. Aromatic Hydrocarbons

Benzene
Butylbenzene
C_3-C_4 Alkylbenzenes
Methylbenzene
Toluene
Xylenes
Isopropenylbenzene

XI. Amines

sec-Butylamine

XII. Sulfides

2-n-Propylthiophene

XIII. Silicon Compounds

Silicon Tetrafluoride
Trimethylsilanol
Various Cyclic and Linear Methylsiloxane Polymers

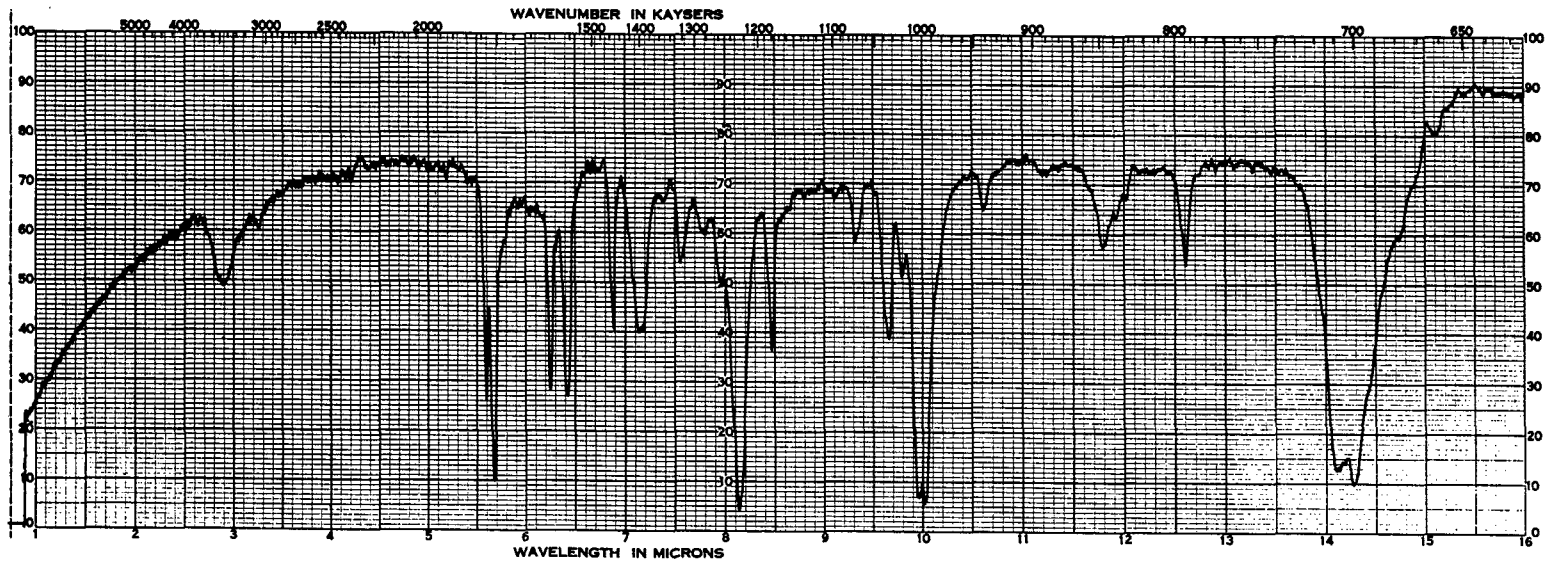


Figure 1. Infrared Spectrum of Crystals from SE 550 Silicone Elastomer (AF 680) (KBr Pellet).

Some gas-off products are identified by compound type only, e.g., alkylbenzene(s), C_4 alkylbenzene(s), or C_4 hydrocarbons. In these cases several homologues or isomers may be present; however, they have not been identified individually.

Some of the gas-off products from silicone-base materials were also calculated collectively. These were the volatile linear and cyclic siloxane polymers (having dimethyl siloxy groups as monomer units) which had been observed in previous gas-off studies (refs. 1,2,3). Although separate peaks are noted in the gas chromatograms (Appendix III), these volatile silicones are listed collectively in the tables of gas-off data (Appendix II) as silicone oil.

Two thiophene derivatives, 2-n-propylthiophene and an uncharacterized weaker compound, were tentatively identified from GLC-fast-scan mass spectrometric measurements of the gas-off products of AF 761. Due to the complexity of the gas chromatographic pattern, the assignment is listed as tentative. We would suggest verifying the identification by using a gas chromatographic detector, such as a flame photometric or Coulson conductivity detector, which would be specific for sulfur-containing compounds.

C. CONCLUSIONS AND RECOMMENDATIONS

The use of thermogravimetric methods (TGA) and water loss data is a good procedure for pre-screening candidate materials. However, the major limitation encountered in this program in applying these techniques for rapid screening is the length of test time (24 hours plus set-up and clean-up time) required for the present test schedule. Ideally, rapid screening should be accomplished in 6-8 hours.

We recommend that considerations be given:

(a) To reduce the time from 4 hours to 1 hour or less to reach isothermal conditions (68°C).

(b) To reduce total test time from a maximum of 24 hours to 6 or 8 hours.

If a constant weight condition for most samples is not reached in 6-8 hours at 68°C, the isothermal test temperature should be raised to compensate for the shorter test period.

SECTION III

THERMAL DECOMPOSITION PRODUCTS OF CARBOXYNITROSO RUBBER (CNR)

Carboxynitroso rubbers (CNR) are potentially valuable materials for use as nonflammable coatings in manned space vehicles. In addition to nonflammability, however, consideration must be given to the possibility of the material thermally degrading to toxic products. For this reason, the thermal degradation of a series of carboxynitroso rubbers was studied by thermogravimetry (TGA). One (MSC-1549) of this series was selected by the Air Force for additional testing, and decomposition products were characterized.

A. THERMOGRAVIMETRIC ANALYSIS

The TGA patterns for the series of carboxynitroso rubbers are shown in Figures 113 to 127 in Appendix IV. Instrument conditions used in the study were as follows:

Instrument - Aminco Thermograv
Sample Weight - 0.1000 gram
Atmosphere - Air (0.04 CFH)
Heating Rate - 5.4°C/min (23°C to 348°C)

Measured weight losses and weight of residue are reported in Table VI.

One feature appearing in Figures 114, 115, 116, 117, 119, and 120 should be noted. The "spike" occurring in the temperature range of 270-310°C results from a sudden downward force applied to the sample crucible by the expanding gases produced in the extremely rapid decomposition of the rubber. The magnitude of spike is inversely proportional to the residue content.

B. IDENTIFICATION OF DECOMPOSITION PRODUCTS

The thermogravimetric (TGA) pattern of the CNR specimen chosen for additional testing is shown in Figure 120. It should be noted that weight loss at 125°C is minimal, weight loss at 250°C is moderate, and that, at 350°C almost complete decomposition has occurred. These three temperatures were selected for the product identification study, with initial tests being performed in 5 psia of pure oxygen. Additional tests were made in 5 psia nitrogen and in vacuum at 350°C in order to establish the influence of a non-oxidizing atmosphere.

Table VI

WEIGHT LOSS AND RESIDUES FOR CARBOXYNITROSO RUBBERS

(100 mg Specimens)

CNR Sample	Weight Loss (mg)	Weight of Residue (mg)
AFE 110 Batch A 014 (with filler)	97.8	2.2
Terpolymer Gum - A 014F	100.0	None
Vulcanizate (without filler) 109-1164	99.7	0.3
GS #1 (MSC 1676)	99.4	0.6
AFE-110 - Batch A-014 (MSC 1672)	98.8	1.2
AFE-110-A-006-1103 (MSC 1481)	97.9	2.1
NRC-NA 3310-43-1&1B (MSC 1549)	100.0	None
Supplemental to MSC 1549	100.0	None
MSC 1475 (Temp. Maximum 950°C)	61.7	38.3
MSC 1480 (Temp. Maximum 970°C)	88.7	11.3
MSC 1673	96.9	3.1
MSC 1674	96.4	3.6
MSC 1675	97.0	3.0

1. Decomposition Conditions

Specimens of 500 mg were sealed in all-glass (Pyrex) test chambers having volumes of 500 cc. In the test vessel, as shown in Figure 2, the specimen (A) was sealed by actually fusing the two glass components together (at B) to form a single, completely enclosed envelope. The sample tube was sufficiently long that the sample could be positioned at one end and thus remain cool while the glass was being sealed at the other end.

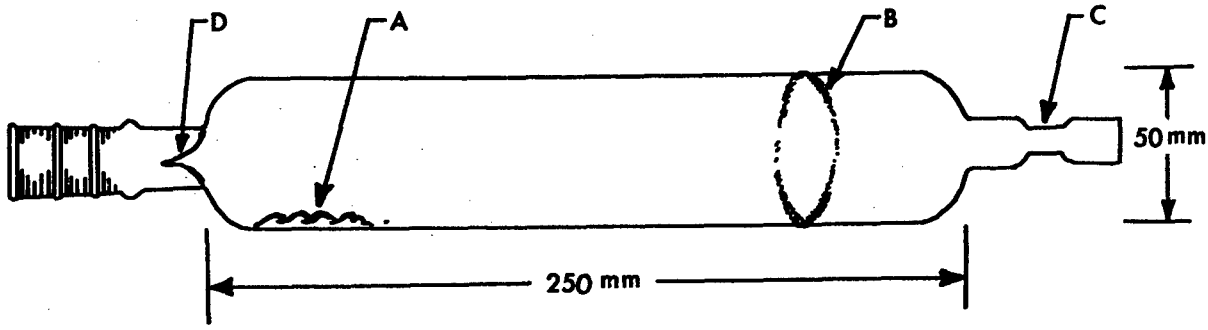
A pure oxygen atmosphere was introduced through tube C and adjusted to a pressure of 5 psia. Tube C was then removed by touching the constricted point with a torch, thus completely encapsulating the sample in glass. The entire chamber with specimen enclosed was placed in an oven at the desired temperature and heated for 1 hour.

Volatile decomposition products were then withdrawn through the standard-taper joint affixed to one end of the vessel. Inside the joint was a glass break-seal, D, which was broken by means of a small steel bar placed inside the tube for that purpose. With the tube in a vertical position, the bar was raised by means of an external magnet and then allowed to fall on the break-seal.

In addition to the glass system, several experiments were performed at 350°C using a stainless steel bomb. The purpose of these experiments was to study the effect of the glass system on the products obtained.

2. Analysis of Decomposition Products

Approximately the same products appear at each of the three temperatures, the chief difference being in the amount formed. Table VII lists the volatile products obtained in the tests along with their mole ratios. Carbon dioxide is taken as unity. Except for the oxides of nitrogen, these same products are formed in a nitrogen atmosphere at 5 psia and also in a complete vacuum. The results of the test, which was performed in a stainless steel bomb at 350°C, are shown in the last column of Table VII. With the glass system, at 350°C large amounts of SiF_4 were obtained. Use of the stainless steel vessel showed that, although a considerable amount of SiF_4 comes from reaction with the glass, much of the SiF_4 is formed from silicon present in the rubber. The presence of a moderately high silicon level in the rubber has been confirmed by emission spectrographic analysis. In the test at 250°C, the amount of silicone tetrafluoride formed is somewhat less, and at 125°C none is detected. Another fluorosilicon compound is formed at 350°C, and it too is lower in the test performed in stainless steel.



A - Sample
B - Seal
C - Gas Inlet Tube
D - Break Seal

Figure 2. Diagram of Break-Seal Flask.

Table VII

VOLATILE DECOMPOSITION PRODUCTS OF CNR MSC 1549

	Components	Mole Ratios of Components Relative to Carbon Dioxide Tested for 1 Hour at 125°C	250°C	350°C	350°C*
Carbon Dioxide	CO_2	1.0	1.0	1.0	1.0
Carbon Monoxide	CO	0.01	0.4	0.5	0.6
Nitrogen Dioxide	NO_2	ND	trace	0.1	0.1
Nitric Oxide	NO	ND	trace	0.02	0.01
Silicone Tetrafluoride	SiF_4	ND	0.2	0.3	0.1
Carbonyl Fluoride	COF_2	0.06	0.2	0.02	0.02
Trifluoromethyl Isocyanate	$CF_3N=C=O$	0.009	0.04	0.04	0.02
Perfluoro- -methyl Methylenimine	$CF_2=N-CF_3$	0.08	0.3	0.05	0.04
Uncharacterized	Silicon Component	ND	ND	0.006	trace
	Perfluorocarbons	0.06	0.005	0.03	0.006
Hexafluorodimethylamine	$(CF_3)_2NH$	ND	trace	ND	0.02
	TGA Results	<1% wt. loss	~10% wt. loss	>95% wt. loss	

*Performed in stainless steel bomb.

The 350°C test performed in the steel bomb shows one other important difference from the one performed in glass. One component, hexafluorodimethylamine, $(CF_3)_2NH$, was detected only in the steel bomb. A trace was found to be formed in glass at 250°C, but none at 350°C.

Carbonyl fluoride is present at all temperatures with a relatively higher amount being observed at 250°C. Perfluoro-*n*-methyl methylenimine (CF_3-N=CF_2) and perfluoromethyl isocyanate (CF_3N=C=O) are also present in relatively higher amounts at 250°C and appear in tests at the other two temperatures as well.

Although temperature does not drastically affect the kinds of products formed, the duration of heating does have a marked influence on certain of the volatile products. If a specimen is heated in the test chamber at 350°C for 72 hours, rather than for 1 hour, no trace of the imine or isocyanate can be detected. Apparently these components are converted to nonvolatile products. This observation can be made regardless of whether the test chamber contains nitrogen or oxygen at 5 psia or whether it has been completely evacuated. If the specimens are heated for only one hour and then analyzed after storing at room temperature for 72 hours, both the isocyanate and imine are present.

Each of the decomposition products was identified by obtaining the mass spectra of the pure components by a combined gas chromatograph-rapid scan mass spectrometer. These data, combined with infrared absorption measurements, are the basis for assignments. Major infrared bands for some of the compounds are shown in Table VIII.

3. Decomposition Mechanisms

An overall reaction for the degradation of nitroso copolymers which yields equimolar amounts of carbonyl fluoride and perfluoro-*n*-methylmethylenimine has been proposed (ref. 4) in equation 1:

$$\left[-CF_2-\overset{CF_3}{\underset{|}{N}}-O-CF_2-\right] \rightarrow CF_2=\overset{CF_3}{\underset{|}{N}} + O=CF_2 \qquad (1)$$

(A) (B) (C)

Table VIII

MAJOR INFRARED ABSORPTION
BONDS FOR DEGRADATION PRODUCTS

Compound	Wavelength (microns)
COF_2	5.20
$CF_3N{=}CF$	5.55
$(CF_3)_2NH$	2.85
$CF_3N{=}C{=}O$	4.35

Minute amounts of water, which are undoubtedly present either on the specimen or in the sample chamber, may cause these compounds to react further (ref. 5), as shown in equations 2 and 3:

$$COF_2 + H_2O \rightarrow CO_2 + 2HF \qquad (2)$$

$$CF_3N{=}CF_2 + H_2O \rightarrow CF_3N{=}C{=}O + 2HF \qquad (3)$$

This accounts for the presence of the perfluoromethyl isocyanate. Hydrofluoric acid formed can react with the glass chamber to form silicon tetrafluoride. The reaction proceeds as in equations 4 and 5 below:

$$SiO_2 + 6HF \rightleftarrows H_2SiF_6 + 2H_2O \qquad (4)$$

$$H_2SiF_6 \rightleftarrows SiF_4 + 2HF \qquad (5)$$

The last reaction proceeds to the right on heating. This may explain the absence of SiF_4 at the lower temperature even though carbonyl fluoride and the imine are present. Hydrofluoric acid may also react with the imine (ref. 4) as indicated in equation 6:

$$CF_3N{=}CF_2 + HF \rightarrow (CF_3)_2NH \qquad (6)$$

The resulting compound, hexafluorodimethylamine, was observed in appreciable amounts only in the test which was performed in the stainless steel bomb. In the glass system this reaction does not proceed, since hydrogen fluoride apparently reacts more readily with the glass.

Much of the CO_2 and CO that was observed is certainly due to decarboxylation of the acid moiety, since these components are formed even in a N_2 atmosphere and in a complete vacuum.

C. CONCLUSIONS

This study has shown that decomposition of the carboxy-nitroso rubber specimen at 125°C is very slight and the major product formed at this temperature is carbon dioxide, presumably due to decarboxylation. Also, a trace amount of carbonyl fluoride is formed. However, at 250°C the specimen loses as much as 10% of its weight, with a significant amount of the highly toxic carbonyl fluoride being formed. A further increase in temperature to 350°C causes complete decomposition, releasing approximately the same products as at 250°C, except that the oxides of nitrogen now appear.

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2. Pustinger, J. V., and F. N. Hodgson, 1967, Identification of Volatile Contaminants of Space Cabin Materials, AMRL-TR-67-58, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio, pp. xvi + 194.

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4. Shultz, A. R., N. Knoll, and G. A. Morneau, "Trifluoronitrosomethane/Tetrafluoroethylene Copolymer: Degradation by Heat and by Radiation," Journal of Polymer Science, Vol. 62, p. 211, 1962.

5. Barr, D. A., and R. N. Hazeldine, "The Synthesis, Propertie and Infrared Spectra of Perfluoroalkyl *iso*Cyanates and Carbamates," Journal of the Chemical Society (London), Vol. 3416, p. 3428, 1956.

APPENDIX I

THERMOGRAVIMETRIC PATTERNS OF CANDIDATE SPACE CABIN MATERIALS

The thermogravimetric analysis (TGA) patterns shown in this appendix were obtained on a Cahn RH Electrobalance. Comparison of the weight loss patterns should be made with care since varying amounts of sample were used to obtain the TGA patterns. The quantity of material used for each TGA measurement is shown on the reproduced pattern.

Water loss curves were computed from data obtained from a Panametrics Model 1000 Hygrometer which was used to monitor continuously the evolution of water.

TGA curves appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

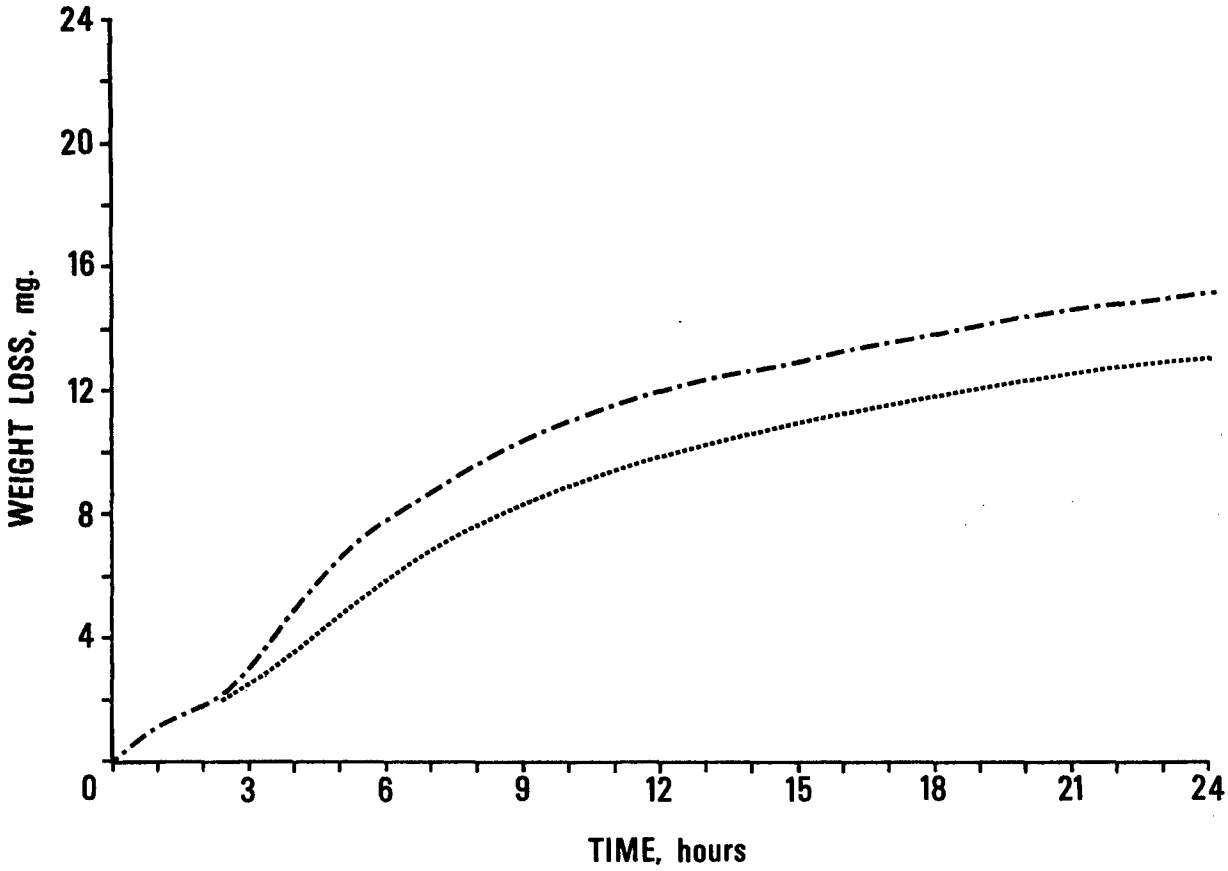

Figure 3. TGA (Upper) and Water Loss (Lower) Curves of Epon 828/Versamid 140 (AF 024).

Specimen Weight - 7.5962 grams

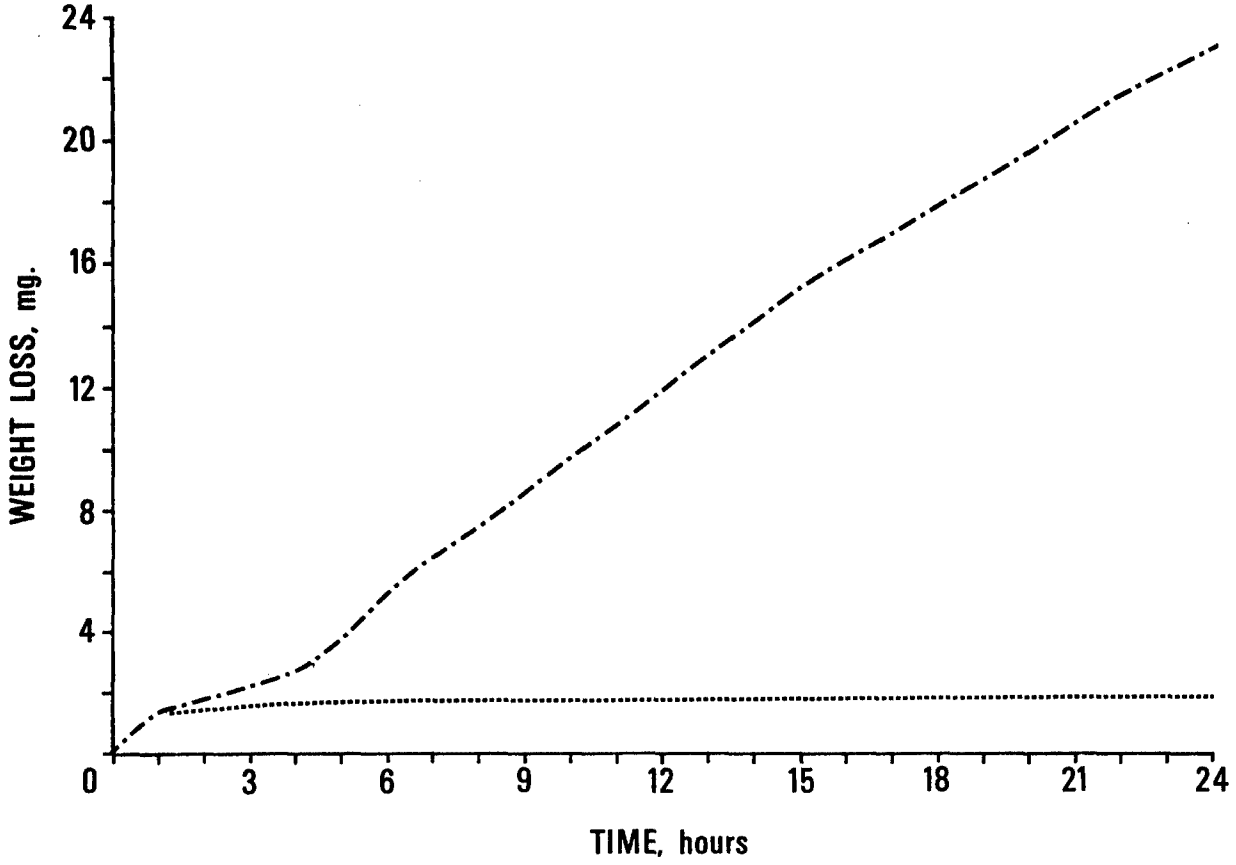

Figure 4. TGA (Upper) and Water Loss (Lower) Curves of Polyester Glass F141 (AF 129).

Specimen Weight - 8.0533 grams

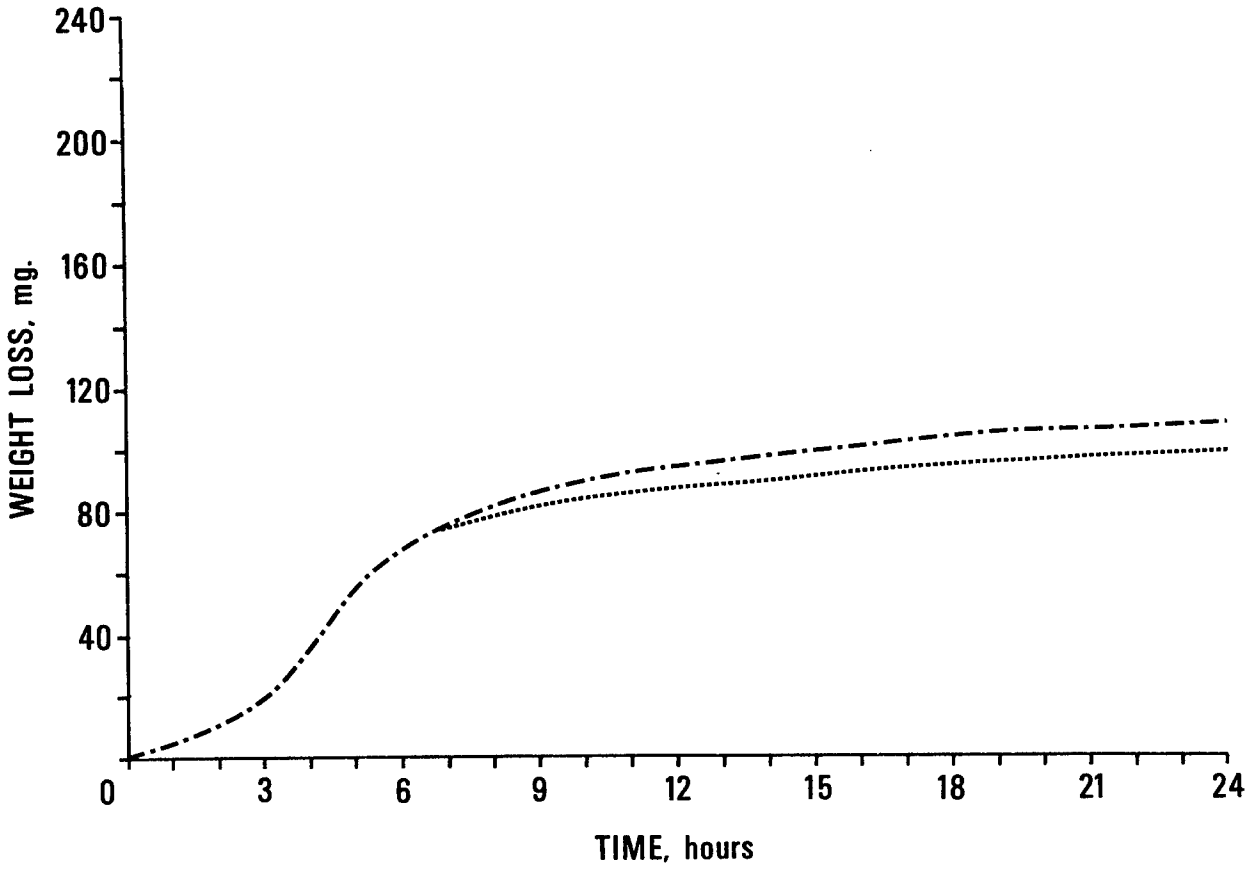

Figure 5. TGA (Upper) and Water Loss (Lower) Curves of Corfil 615 Adhesive (Dried) (AF 225).

Specimen Weight - 8.0939 grams

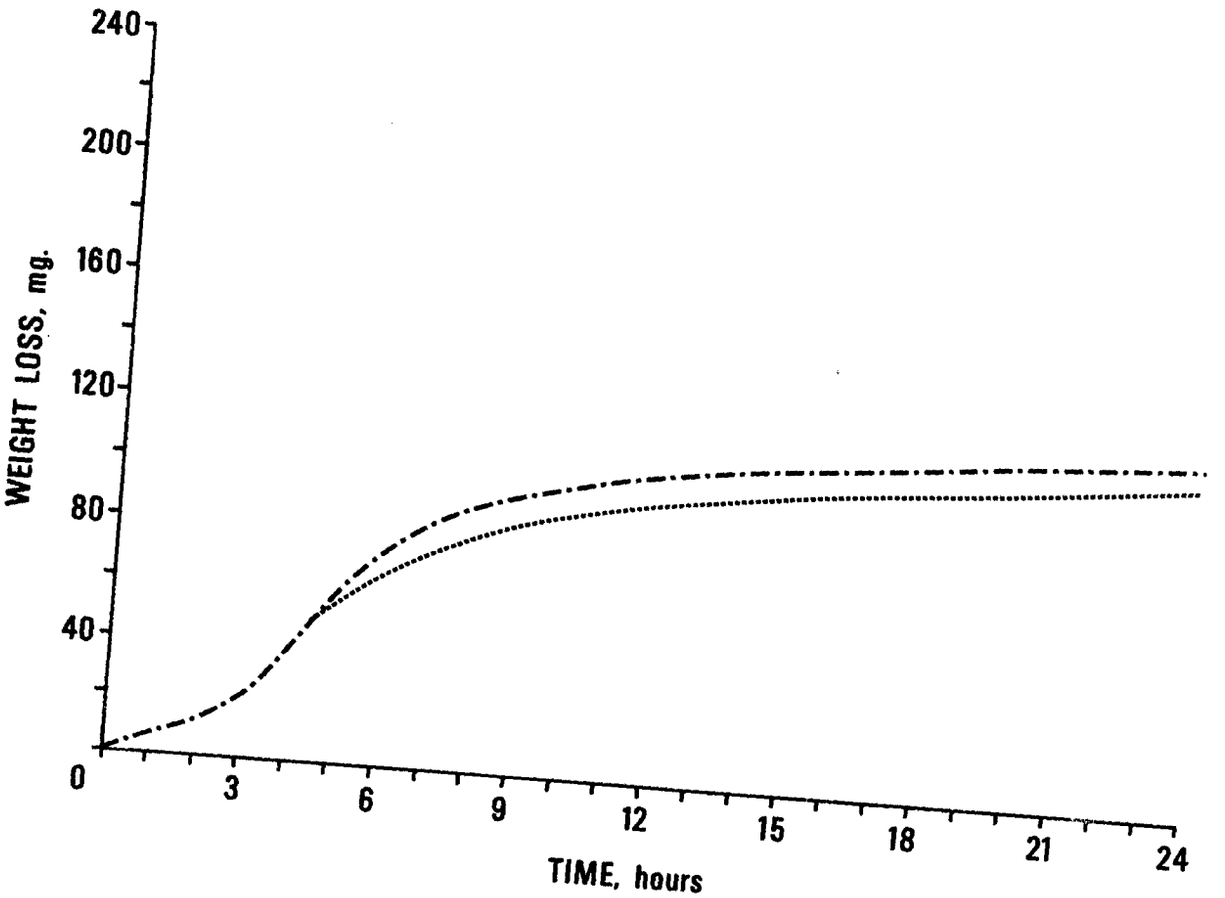

Figure 6. TGA (Upper) and Water Loss (Lower) Curves of Corfil 615 Adhesive (Not Dried) (AF 225). Specimen Weight - 7.8779 grams

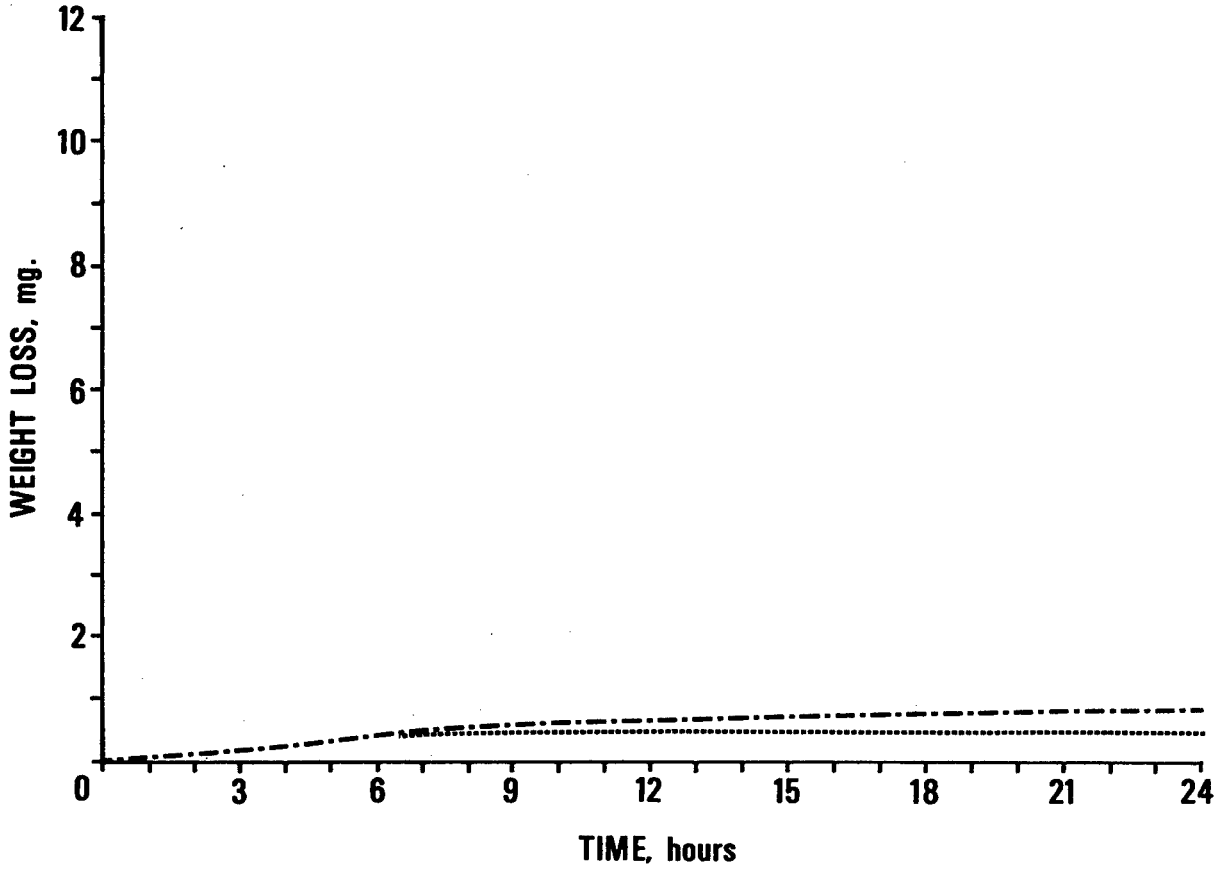

Figure 7. TGA (Upper) and Water Loss (Lower) Curves of Vinylidene Fluoride, Kaynar (AF 250).
Specimen Weight - 10.0140 grams

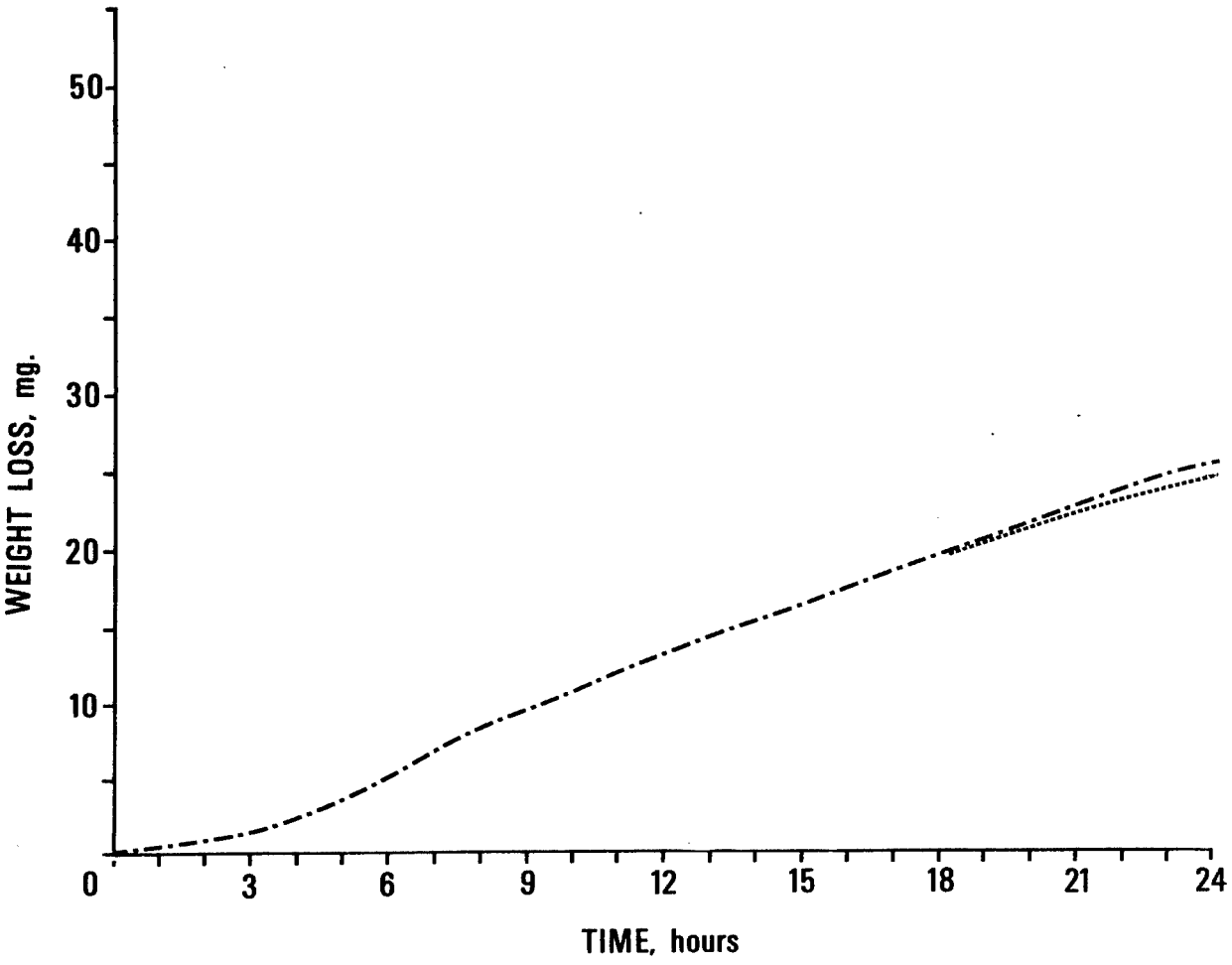

Figure 8. TGA (Upper) and Water Loss (Lower) Curves of Plex 55 Acrylic (AF 256).

Specimen Weight - 10.0859 grams

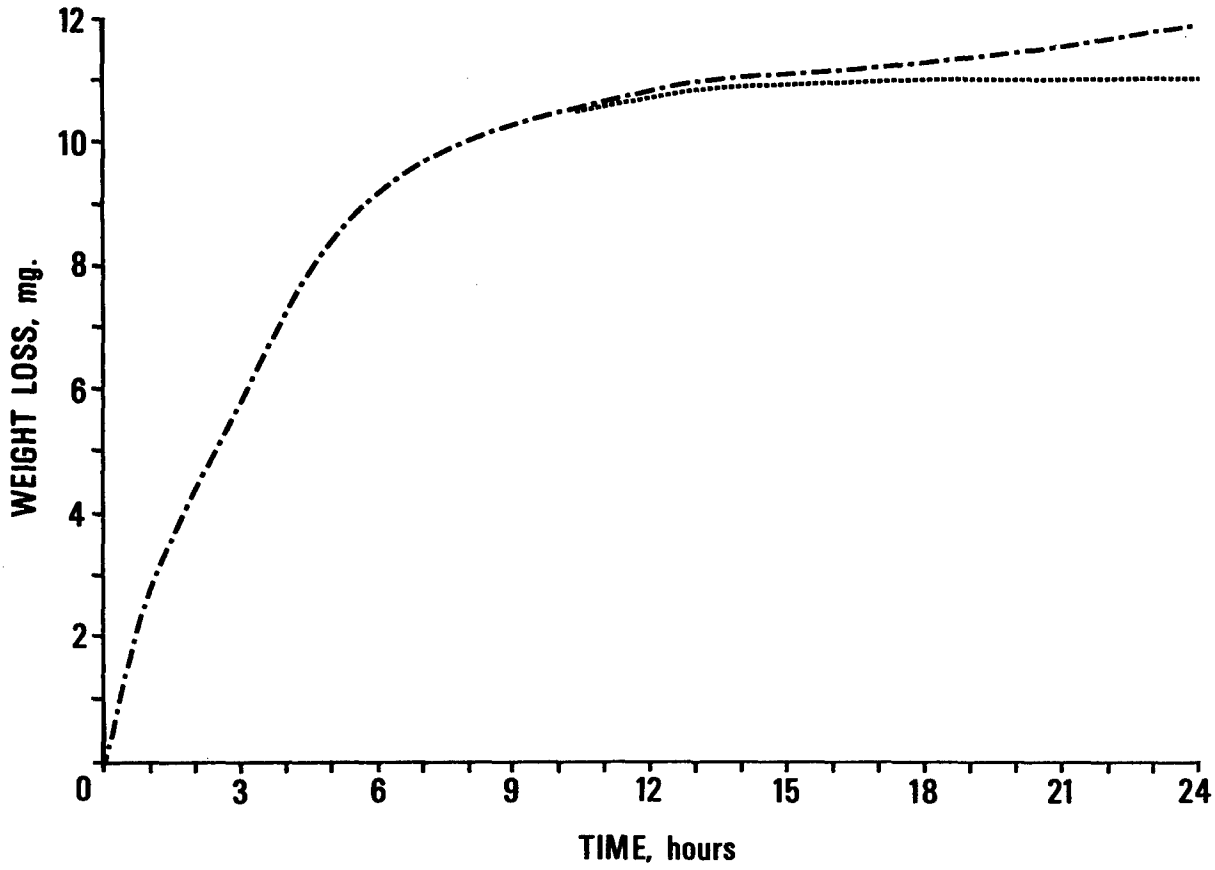

Figure 9. TGA (Upper) and Water Loss (Lower) Curves of Silicone Elastomer Q2-0078 (AF 407).

Specimen Weight - 8.5350 grams

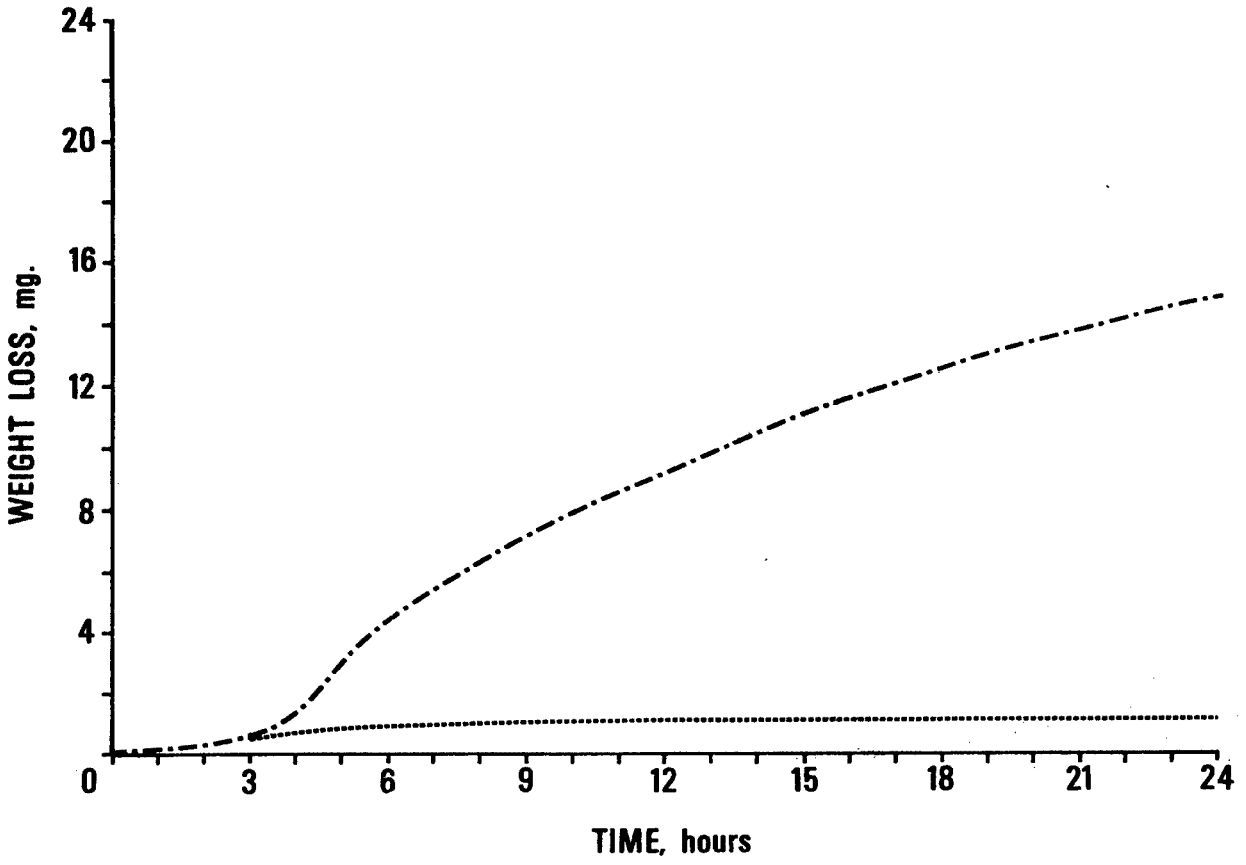

Figure 10. TGA (Upper) and Water Loss (Lower) Curves of Diallyl Phthalate 52 (AF 417).

Specimen Weight - 5.1508 grams

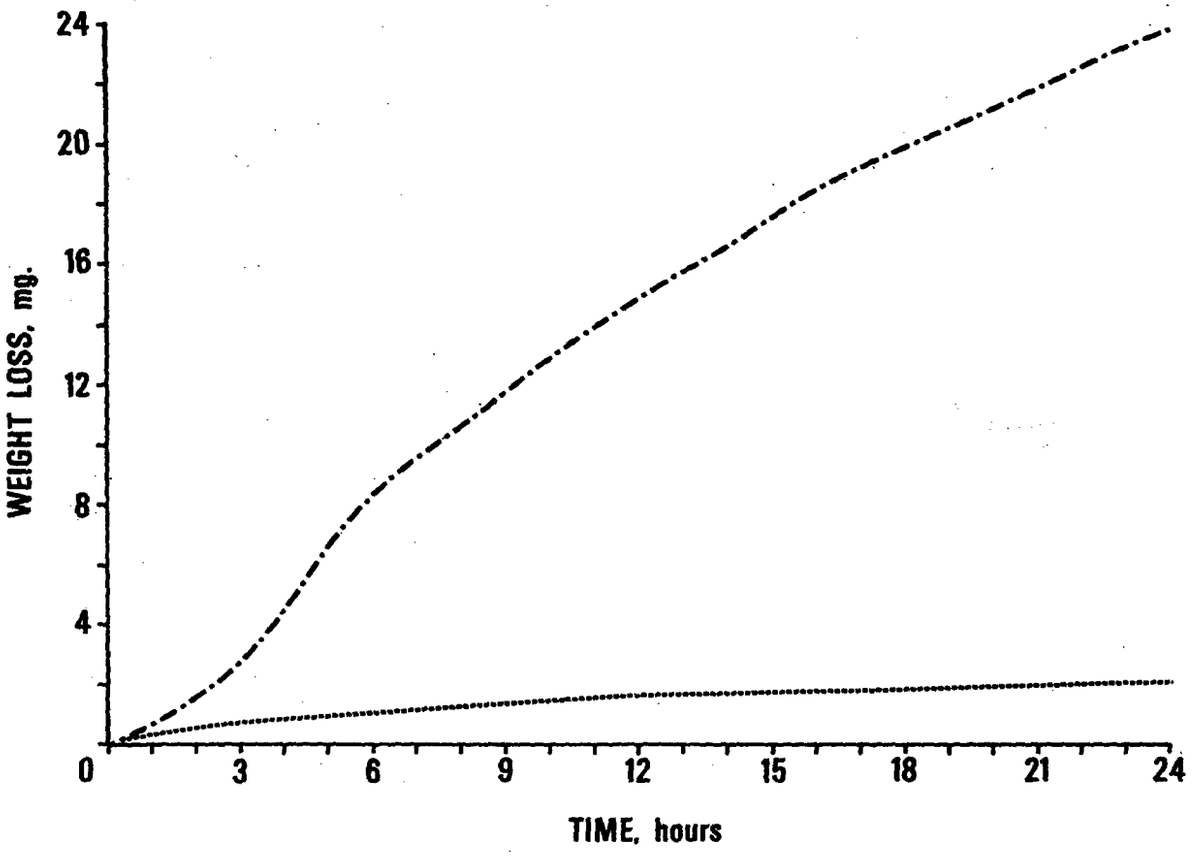

Figure 11. TGA (Upper) and Water Loss (Lower) Curves of Huniseal 1B12 Laminate (AF 465).

Specimen Weight - 6.7777 grams

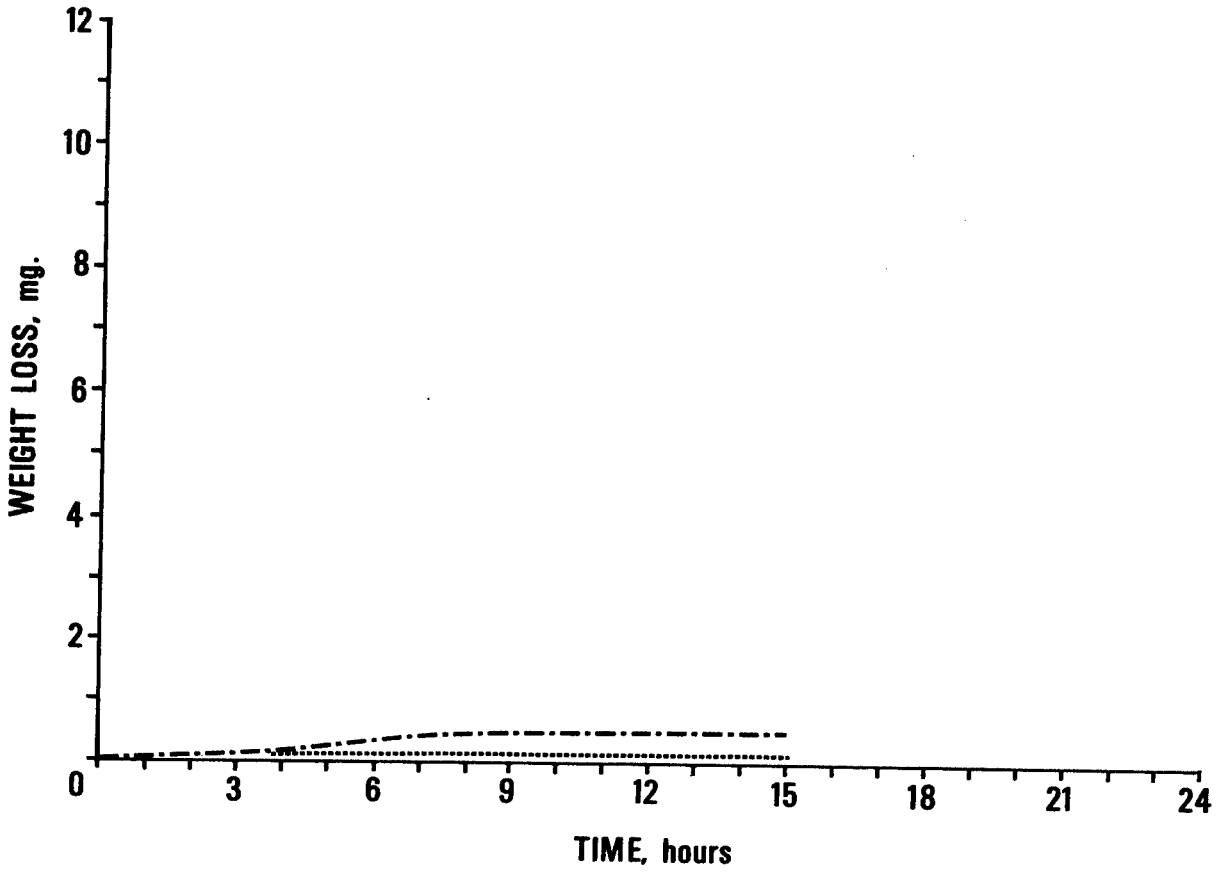

Figure 12. TGA (Upper) and Water Loss (Lower) Curves of Tape-Temp-R-Glass (AF 491).

Specimen Weight - 10.1321 grams

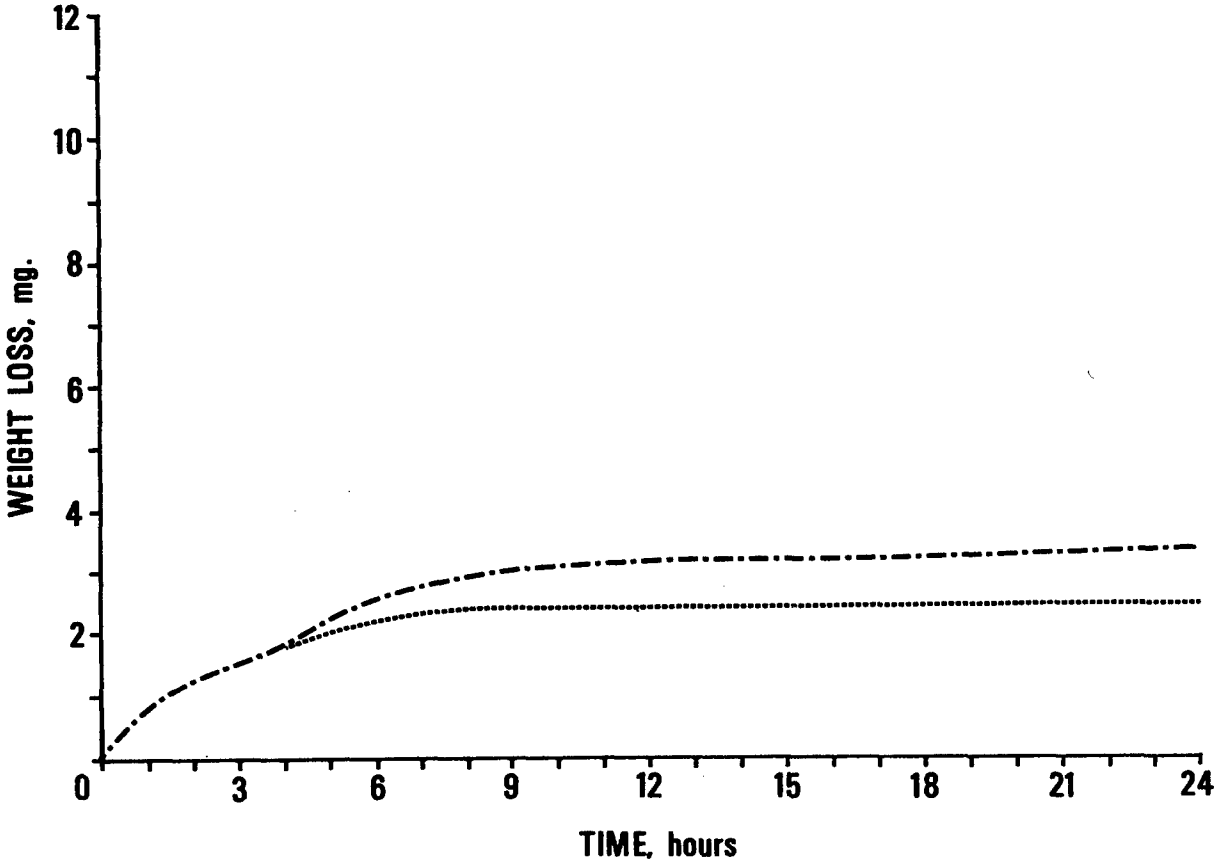

Figure 13. TGA (Upper) and Water Loss (Lower) Curves of Lexan 101-01 (AF 532).

Specimen Weight - 9.9917 grams

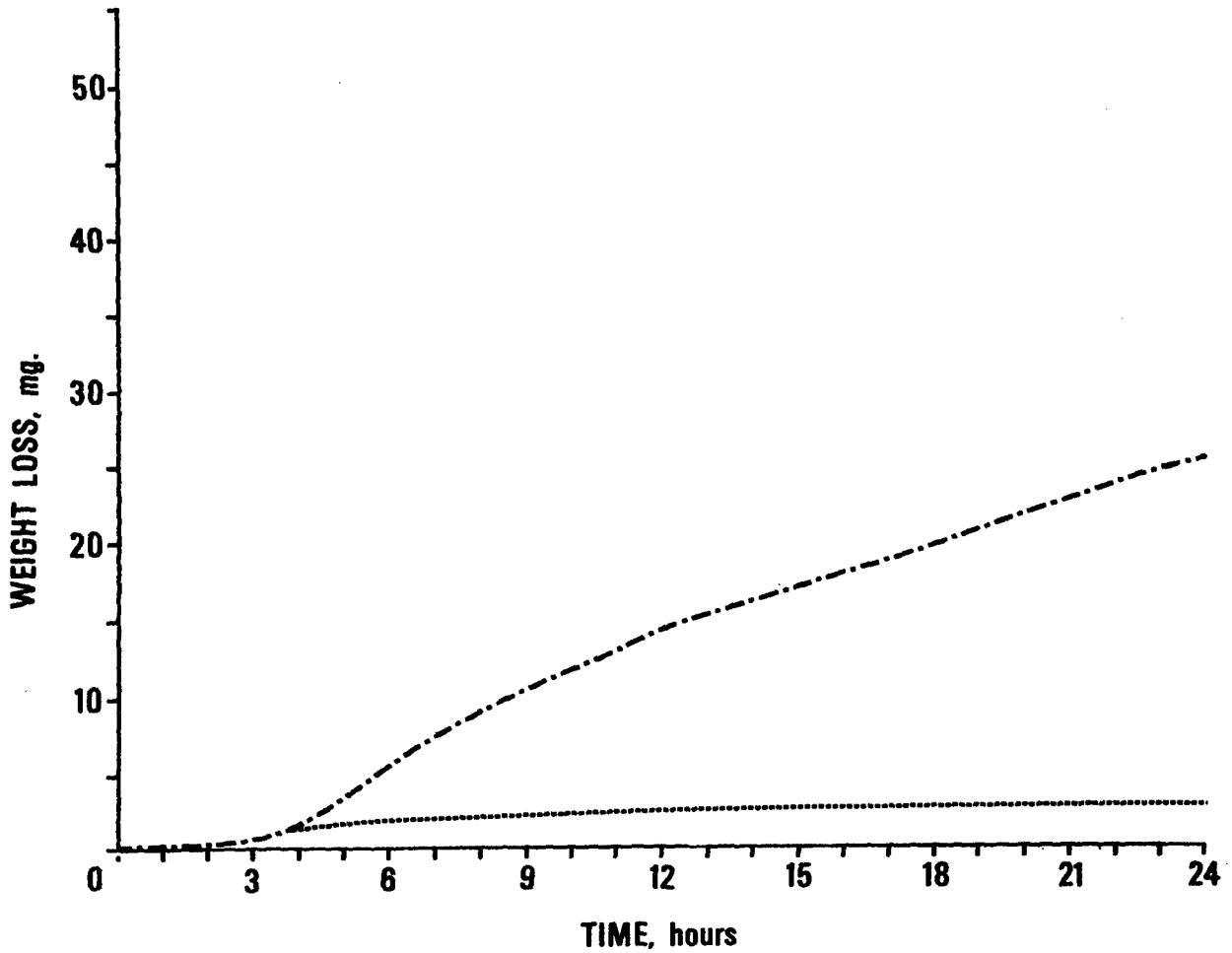

Figure 14. TGA (Upper) and Water Loss (Lower) Curves of Locquic Primer Grade N (AF 616).

Specimen Weight - 0.1783 gram

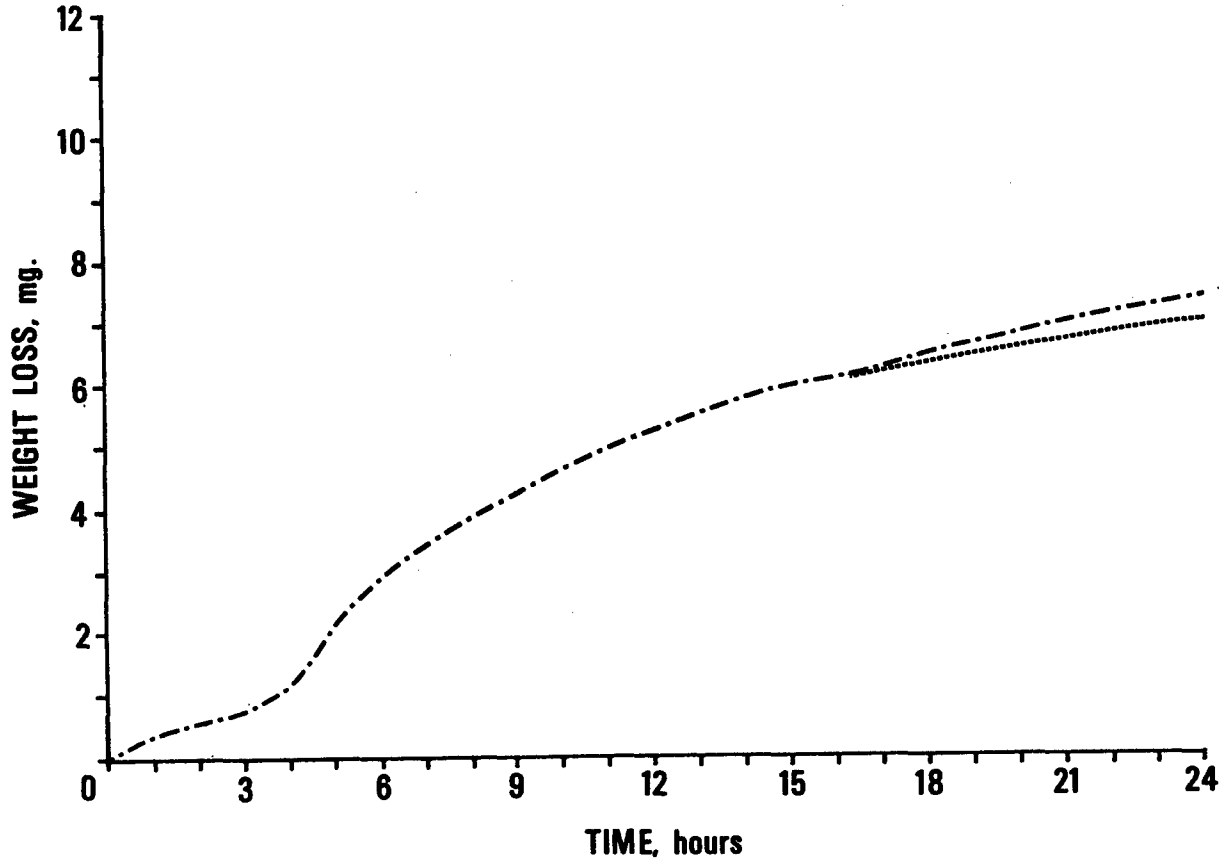

Figure 15. TGA (Upper) and Water Loss (Lower) Curves of A2 Epoxy Adhesive and Activator A (AF 619).

Specimen Weight - 10.2945 grams

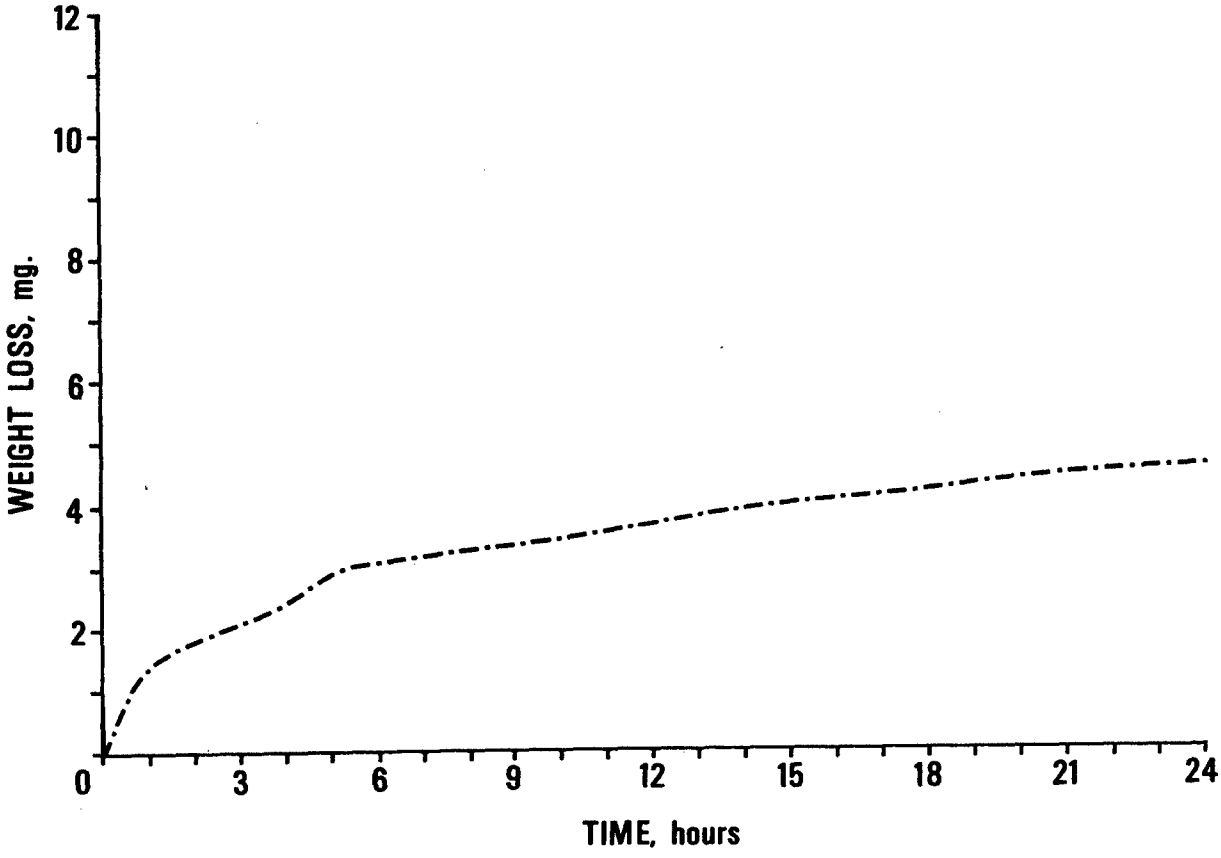

Figure 16. TGA Curve of 1151 Flex Tubing (Silicone Rubber on Glass) (AF 623).

Specimen Weight - 10.9926 grams

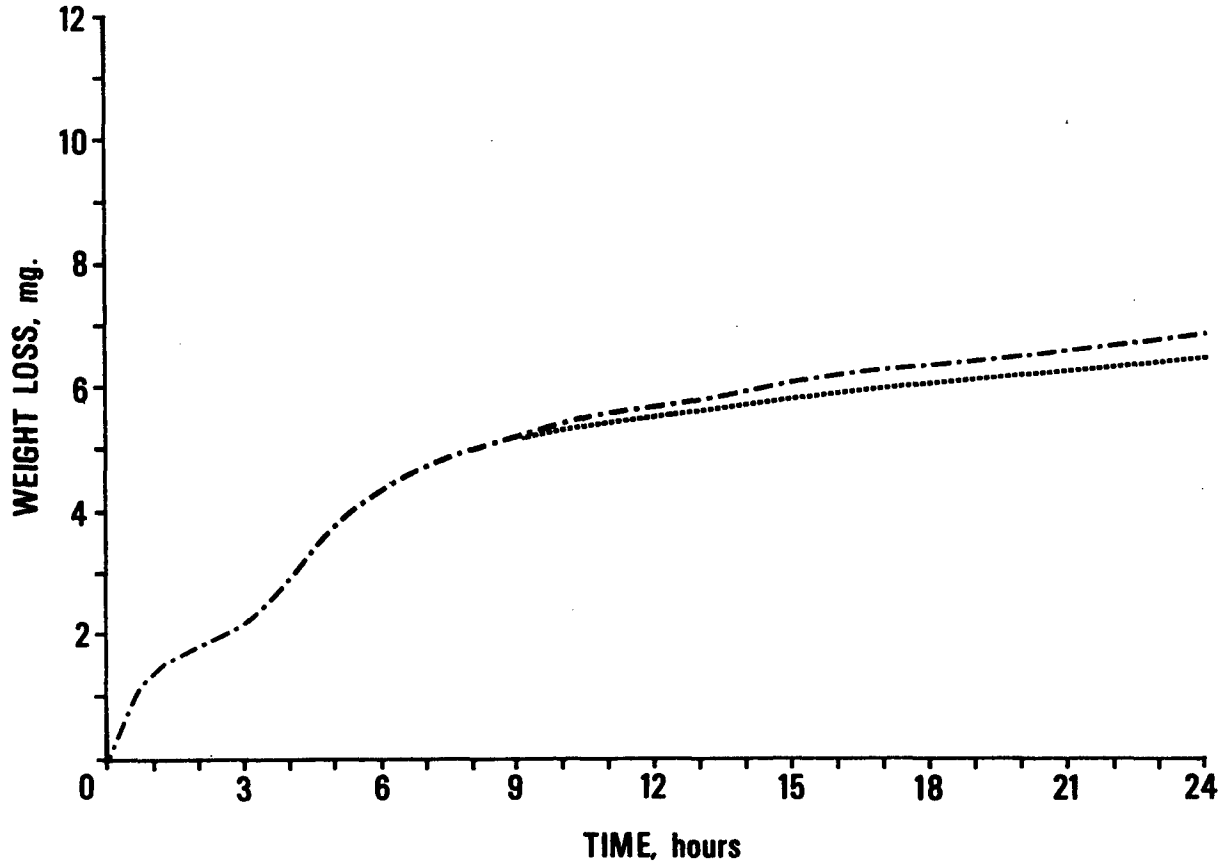

Figure 17. TGA (Upper) and Water Loss (Lower) Curves of CHR 3320 Silicone/Glass (AF 639).
Specimen Weight - 9.6219 grams

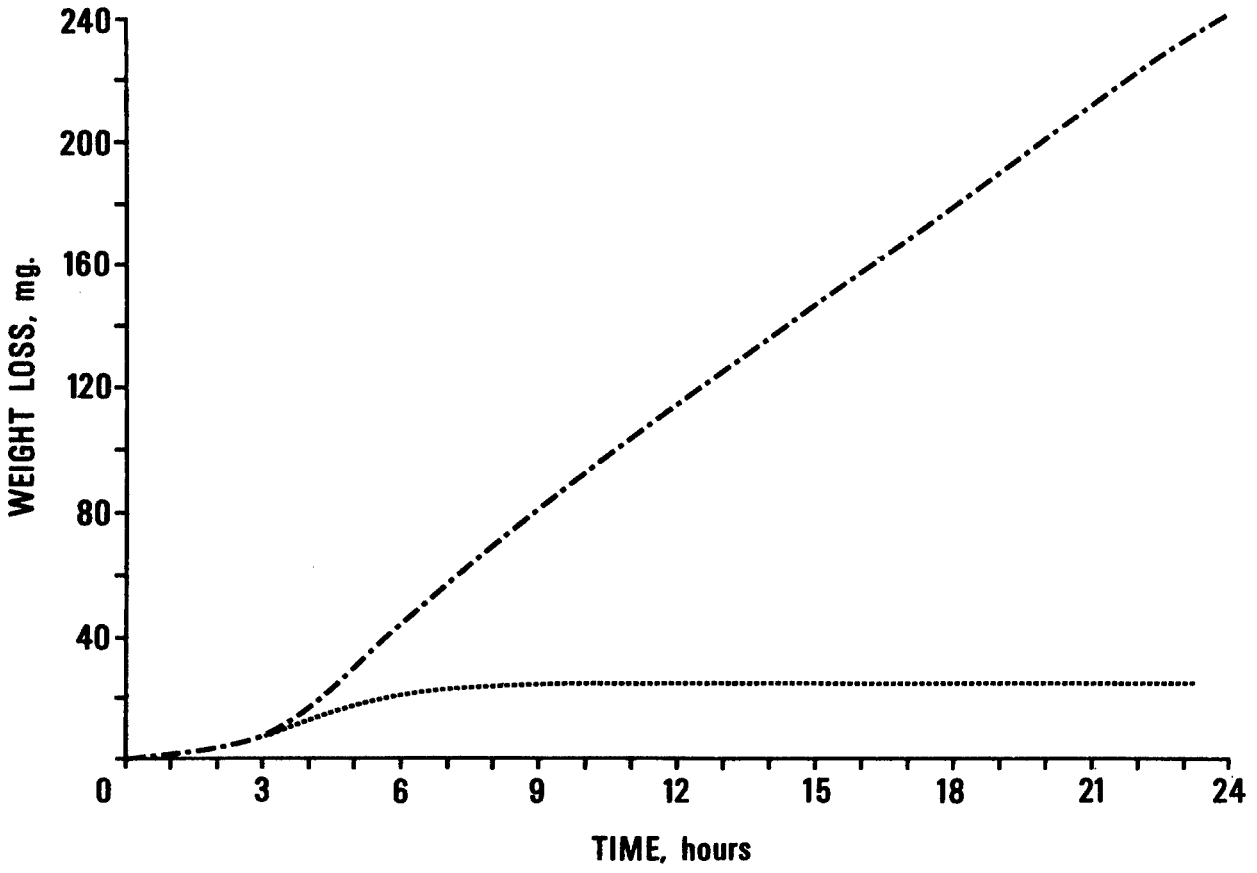

Figure 18. TGA (Upper) and Water Loss (Lower) Curves of 9711 Silicone Rubber (AF 643).

Specimen Weight - 8.3022 grams

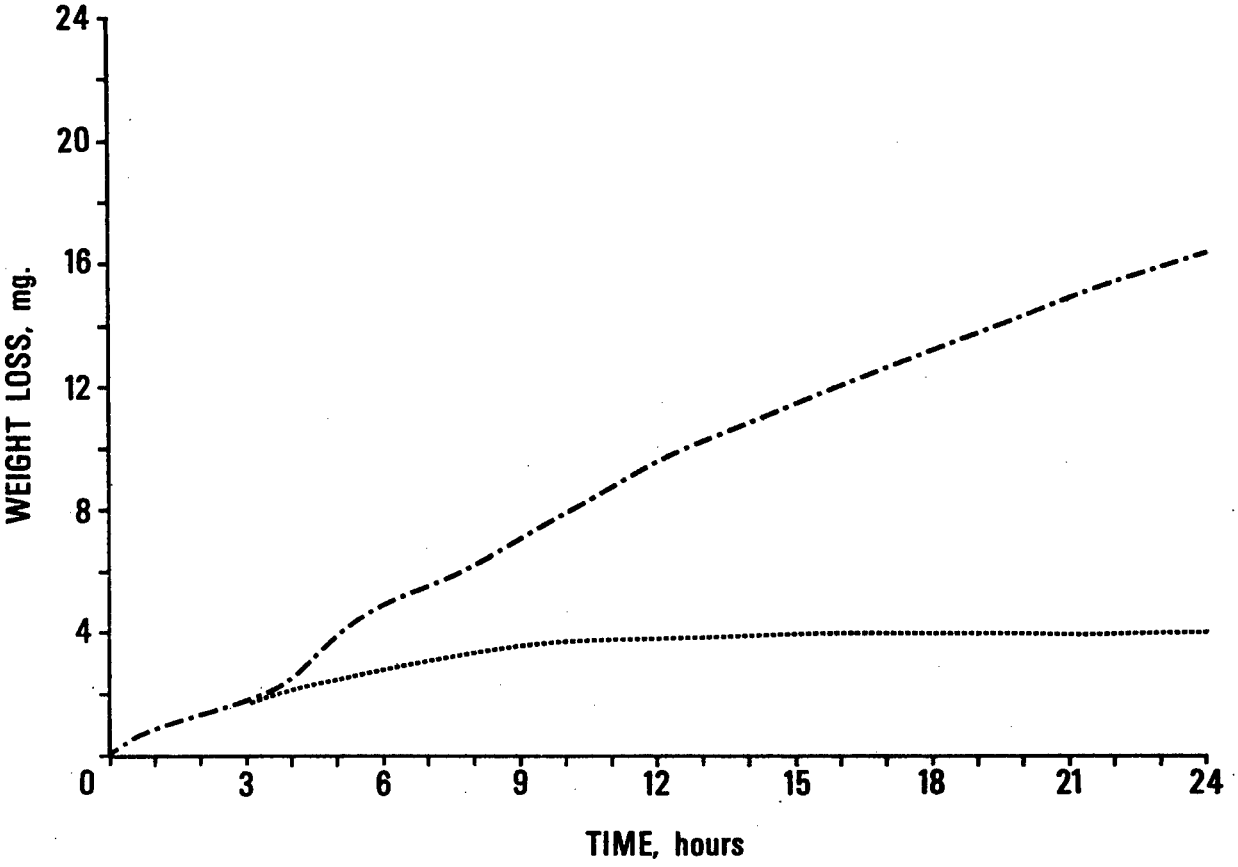

Figure 19. TGA (Upper) and Water Loss (Lower) Curves of DC-5 Lube (AF 645).

Specimen Weight - 11.6142 grams

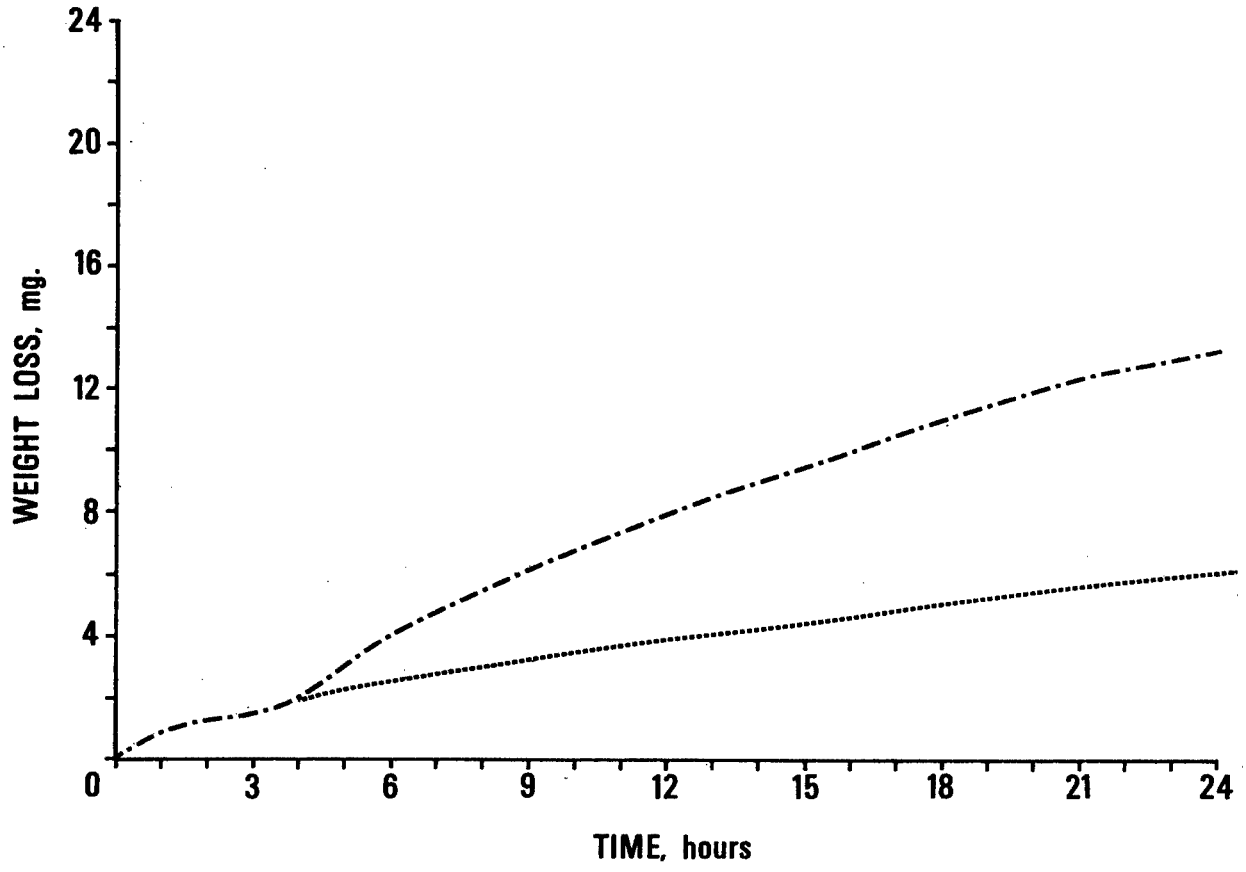

Figure 20. TGA (Upper) and Water Loss (Lower) Curves of DC-33 Light Grease (AF 646).

Specimen Weight - 9.6525 grams

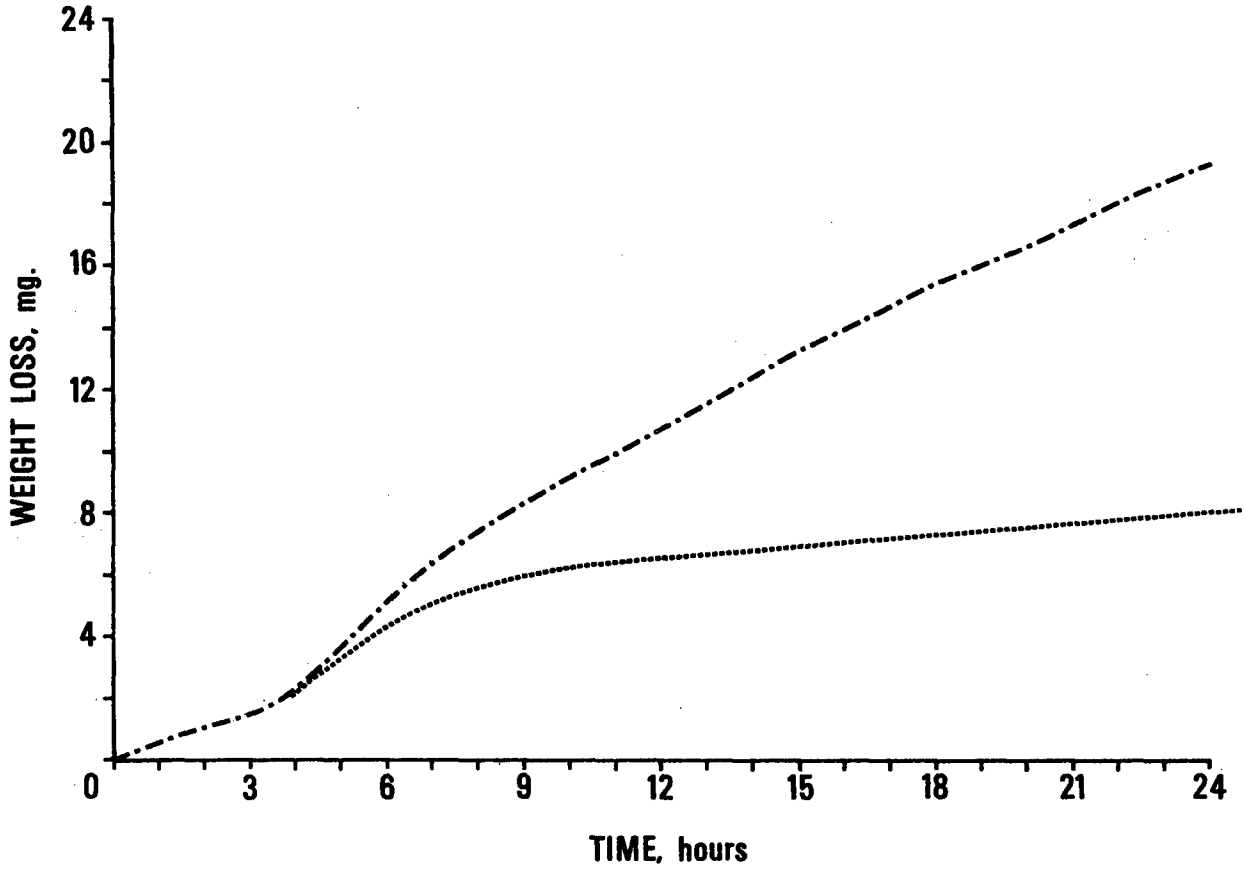

Figure 21. TGA (Upper) and Water Loss (Lower) Curves of DC-33 Medium Grease (AF 647).

Specimen Weight - 10.0667 grams

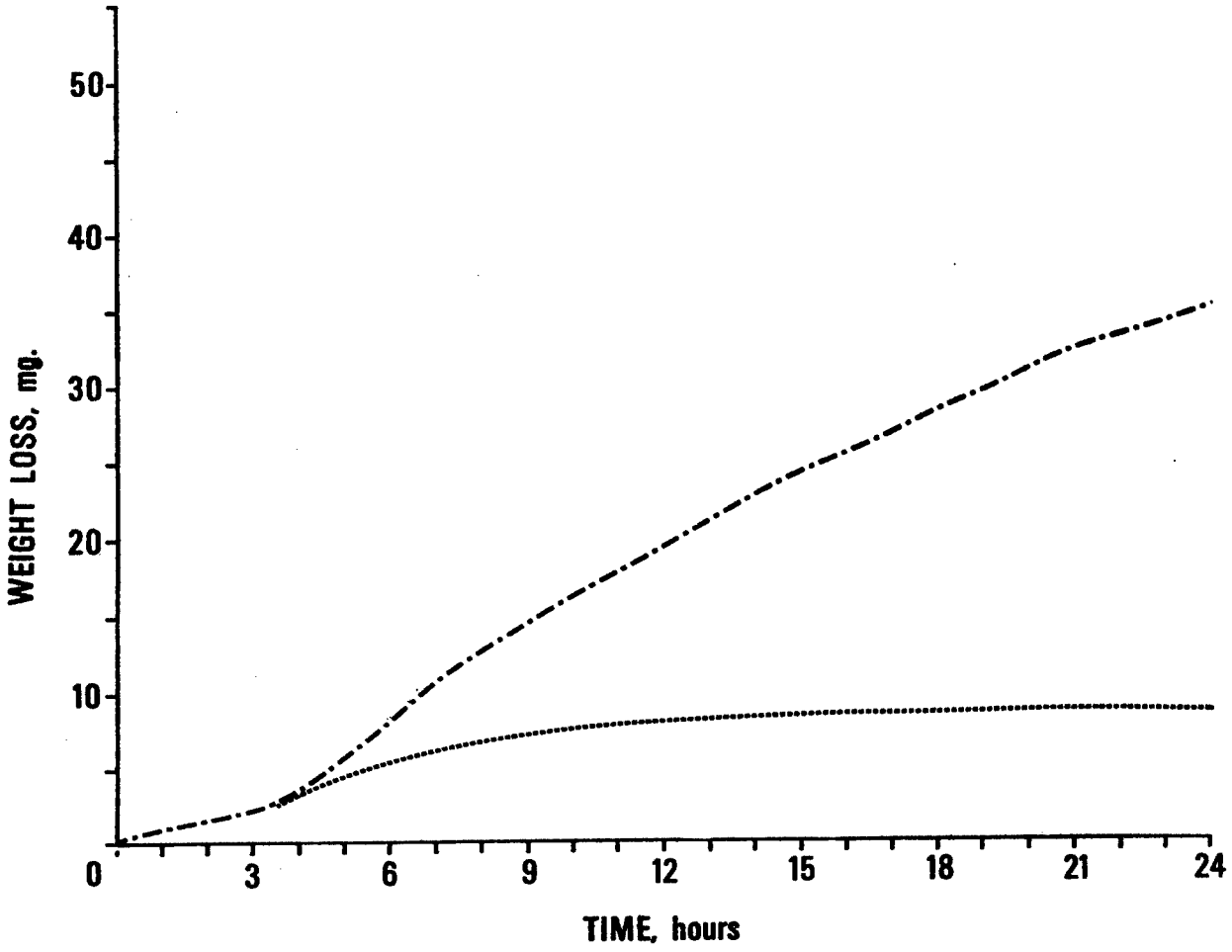

Figure 22. TGA (Upper) and Water Loss (Lower) Curves of DC-33 Heavy Grease (AF 648).

Specimen Weight - 9.1242 grams

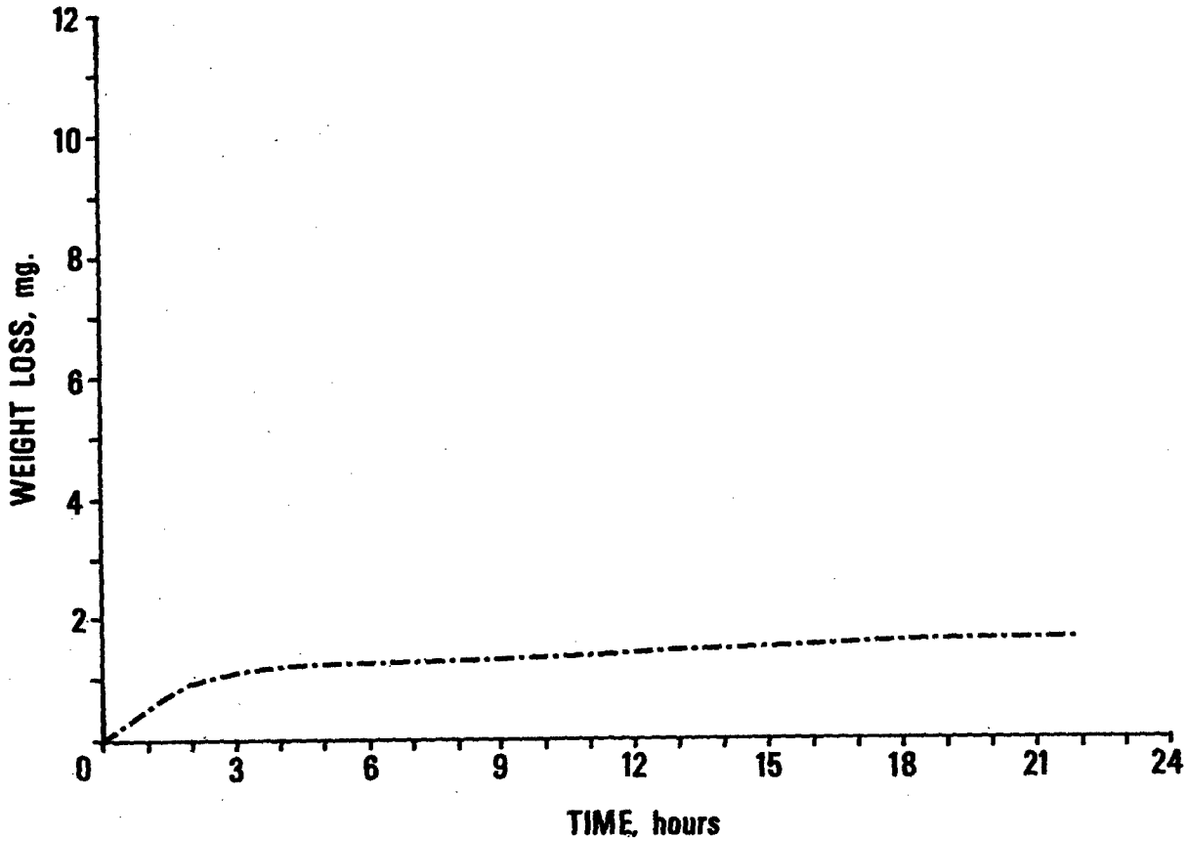

Figure 23. TGA Curve of DC-510 Lube (Silicon) (AF 649).

Specimen Weight - 6.9971 grams

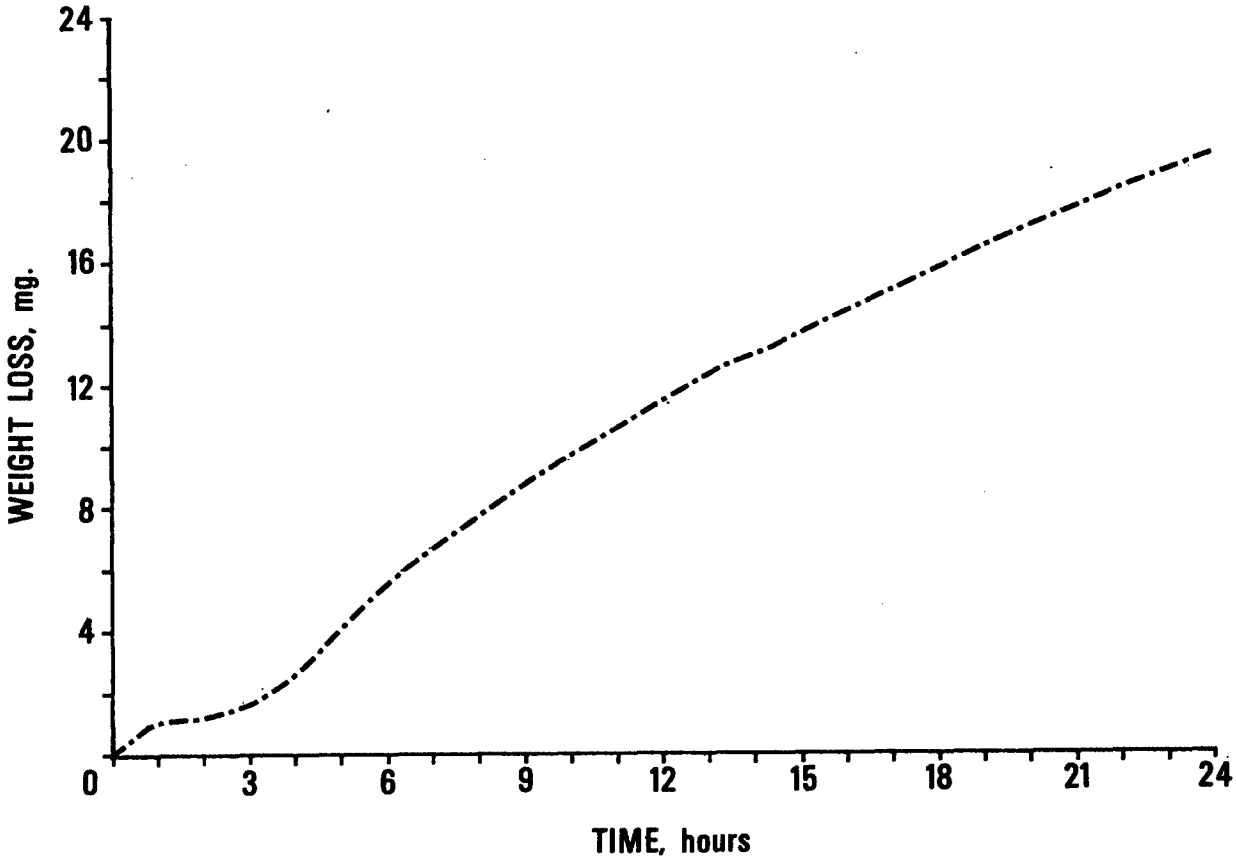

Figure 24. TGA Curve of RTV 501 Potting Compound (AF 653).

Specimen Weight - 10.0607 grams

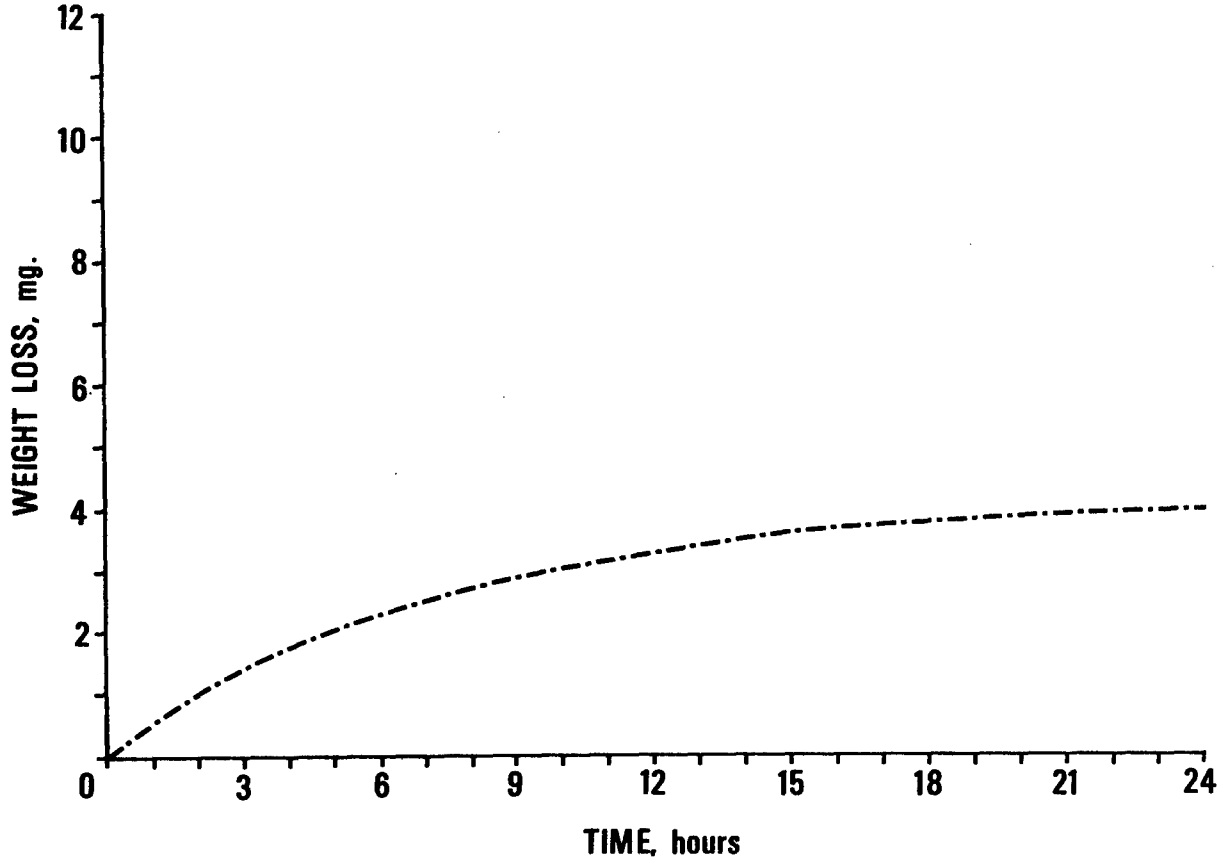

Figure 25. TGA Curve of Silastic 8164 (AF 658).

Specimen Weight - 10.0965 grams

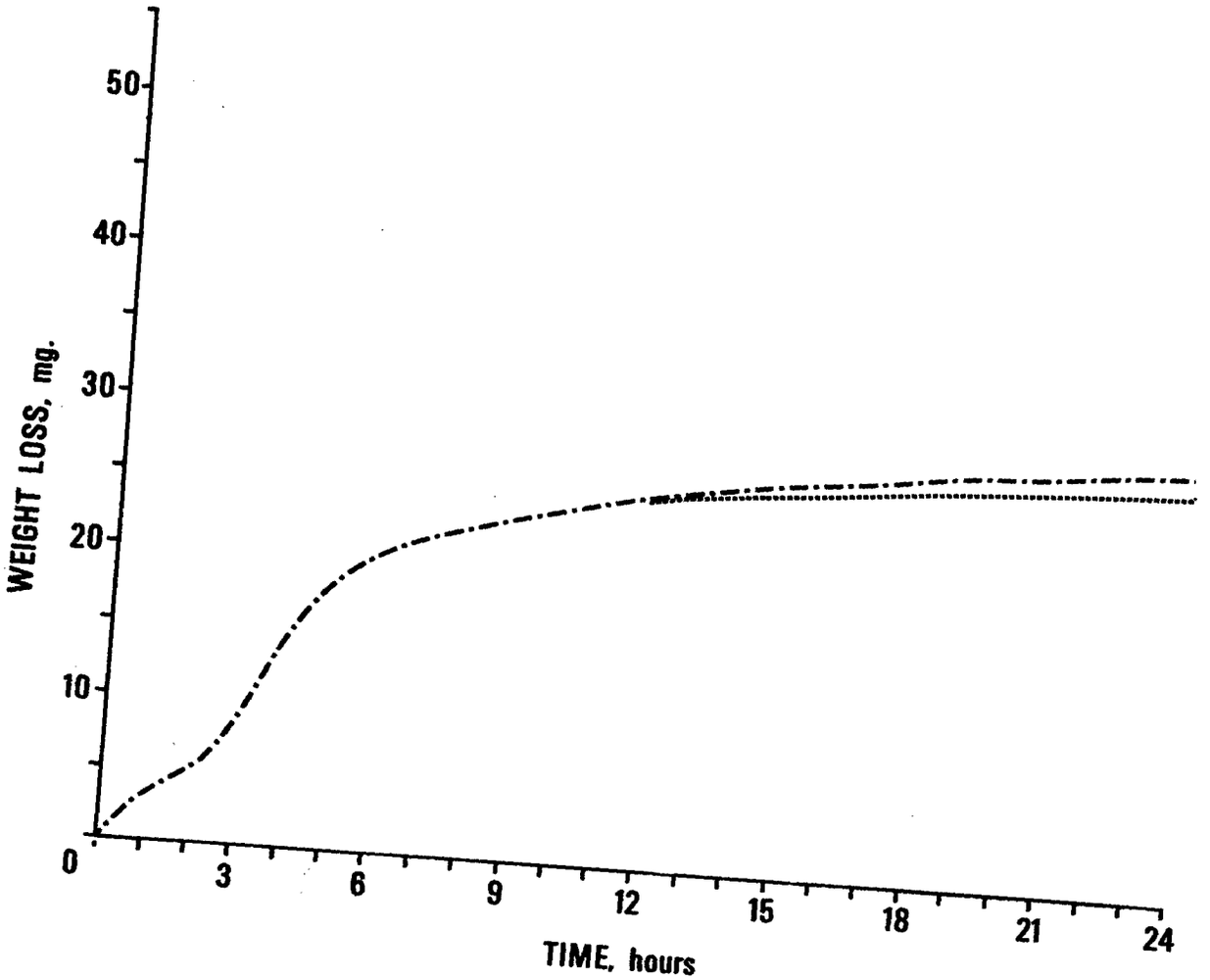

Figure 26. TGA (Upper) and Water Loss (Lower) Curves of Stycast 1090/Cat 9 (AF 669-9).
Specimen Weight - 7.2966 grams

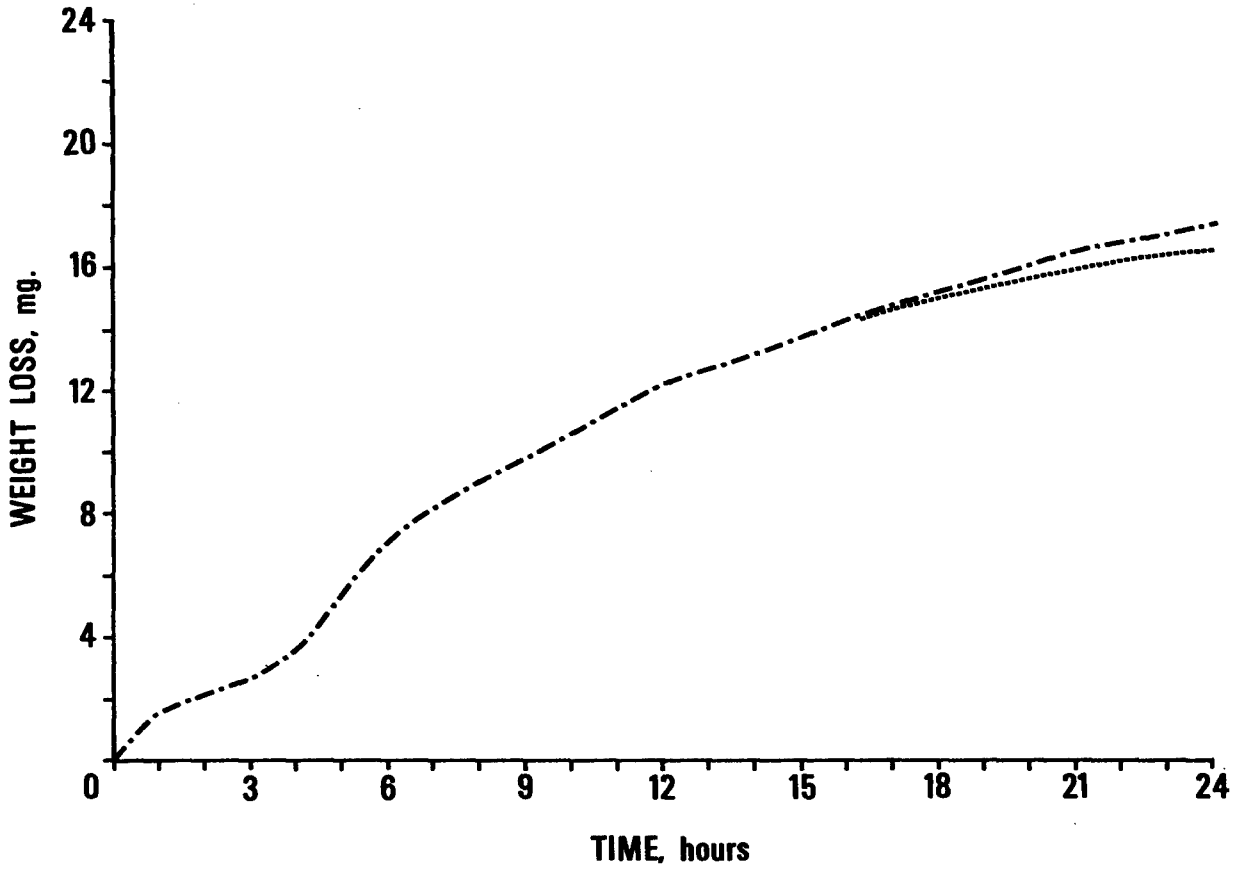

Figure 27. TGA (Upper) and Water Loss (Lower) Curves of Stycast 1090/Cat 11 (AF 669-11).

Specimen Weight - 10.7313 grams

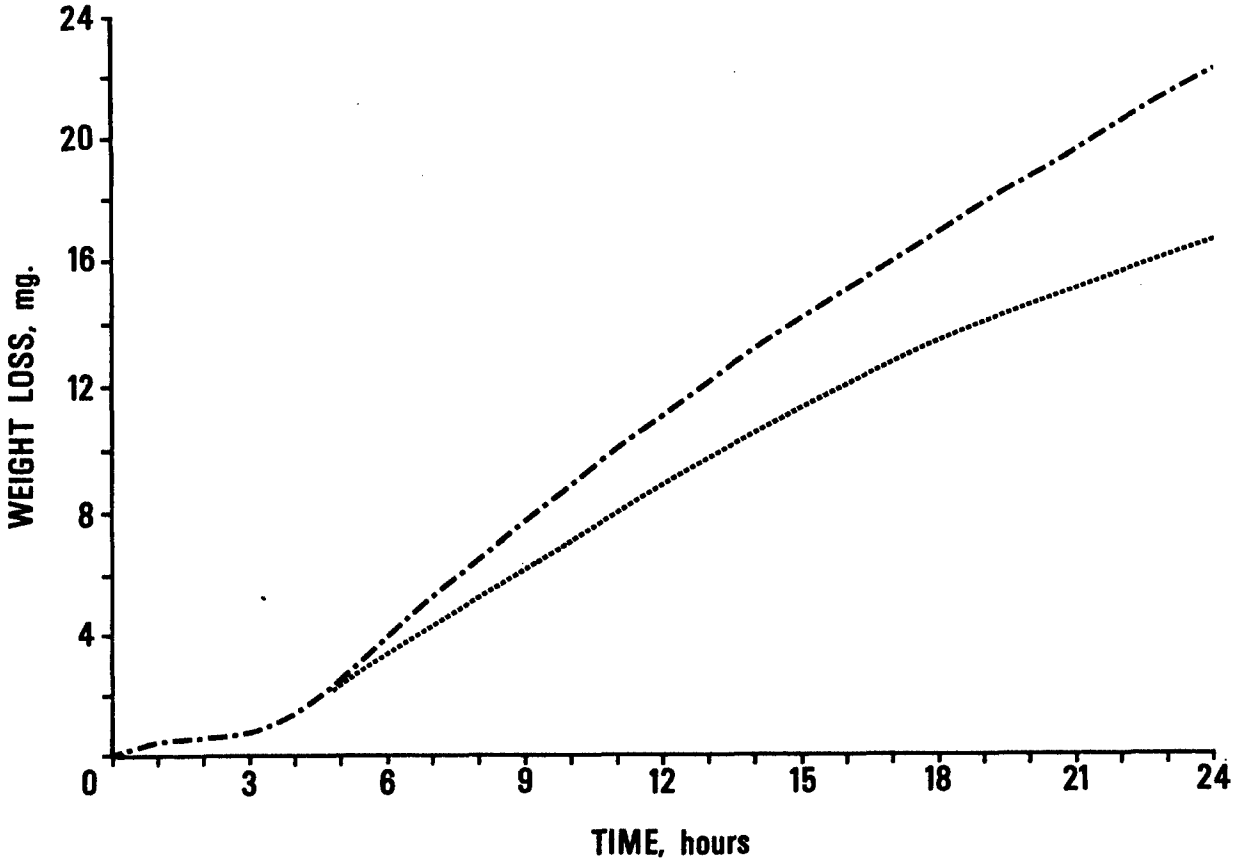

Figure 28. TGA (Upper) and Water Loss (Lower) Curves of Urelane 5712 (AF 675).

Specimen Weight - 10.6037 grams

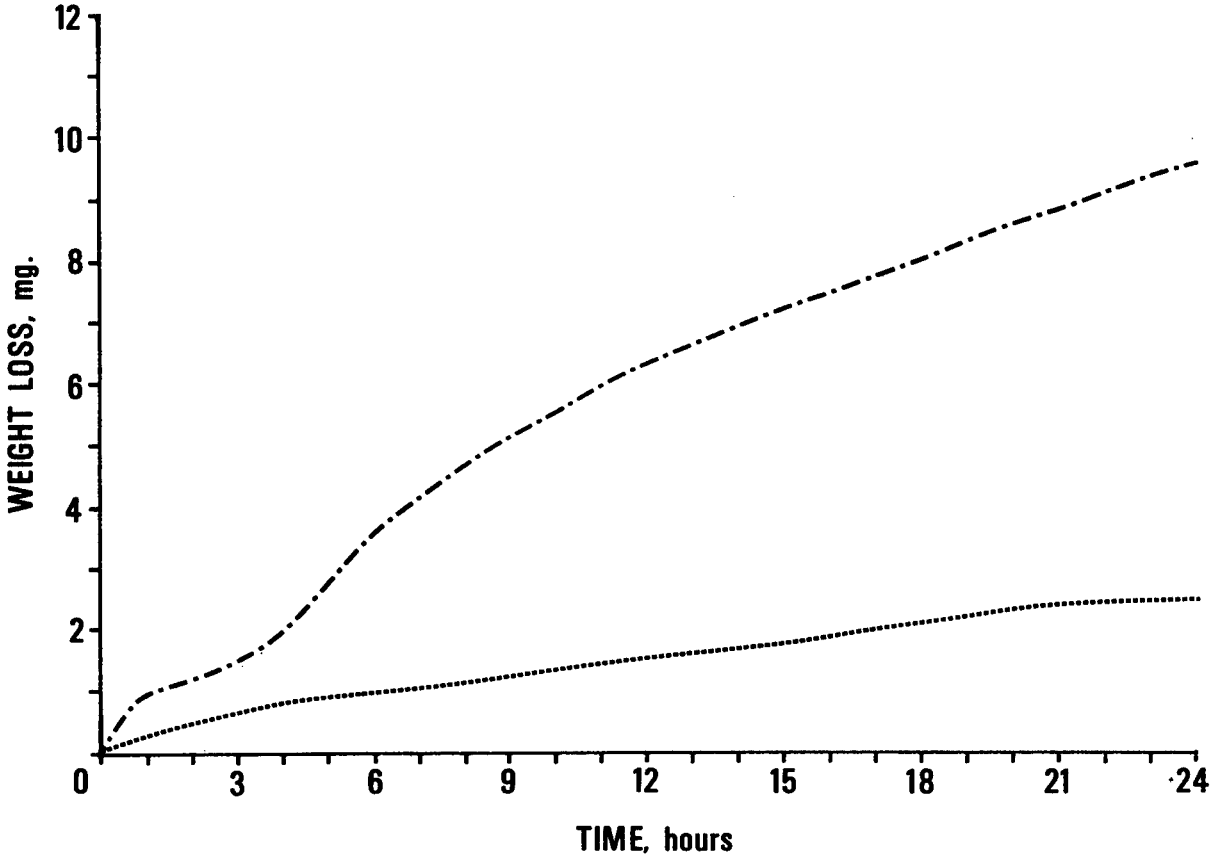

Figure 29. TGA (Upper) and Water Loss (Lower) Curves of RTV-511 Silicone Rubber (AF 678).

Specimen Weight - 9.6593 grams

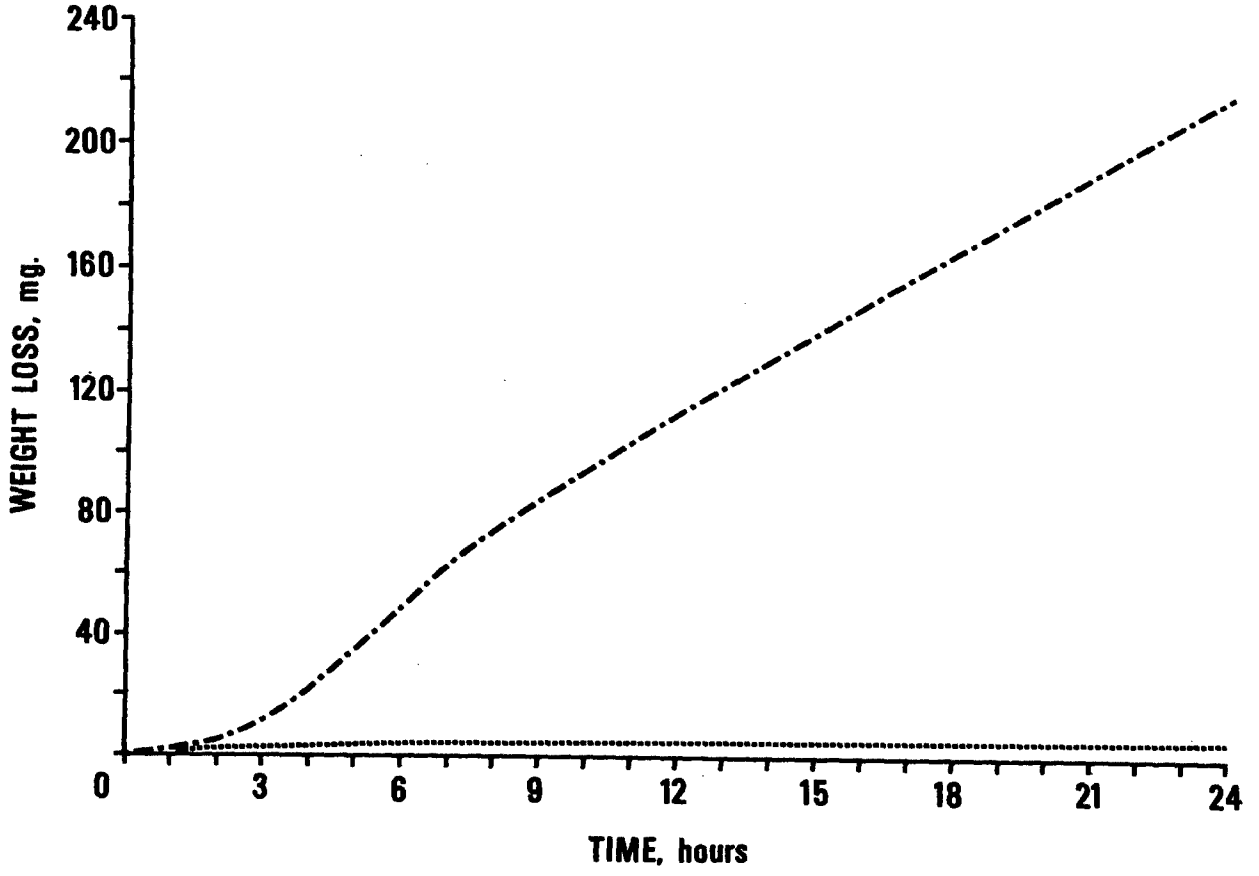

Figure 30. TGA (Upper) and Water Loss (Lower) Curves of SE 550 Silicone Elastomer (AF 680).
Specimen Weight - 10.1077 grams

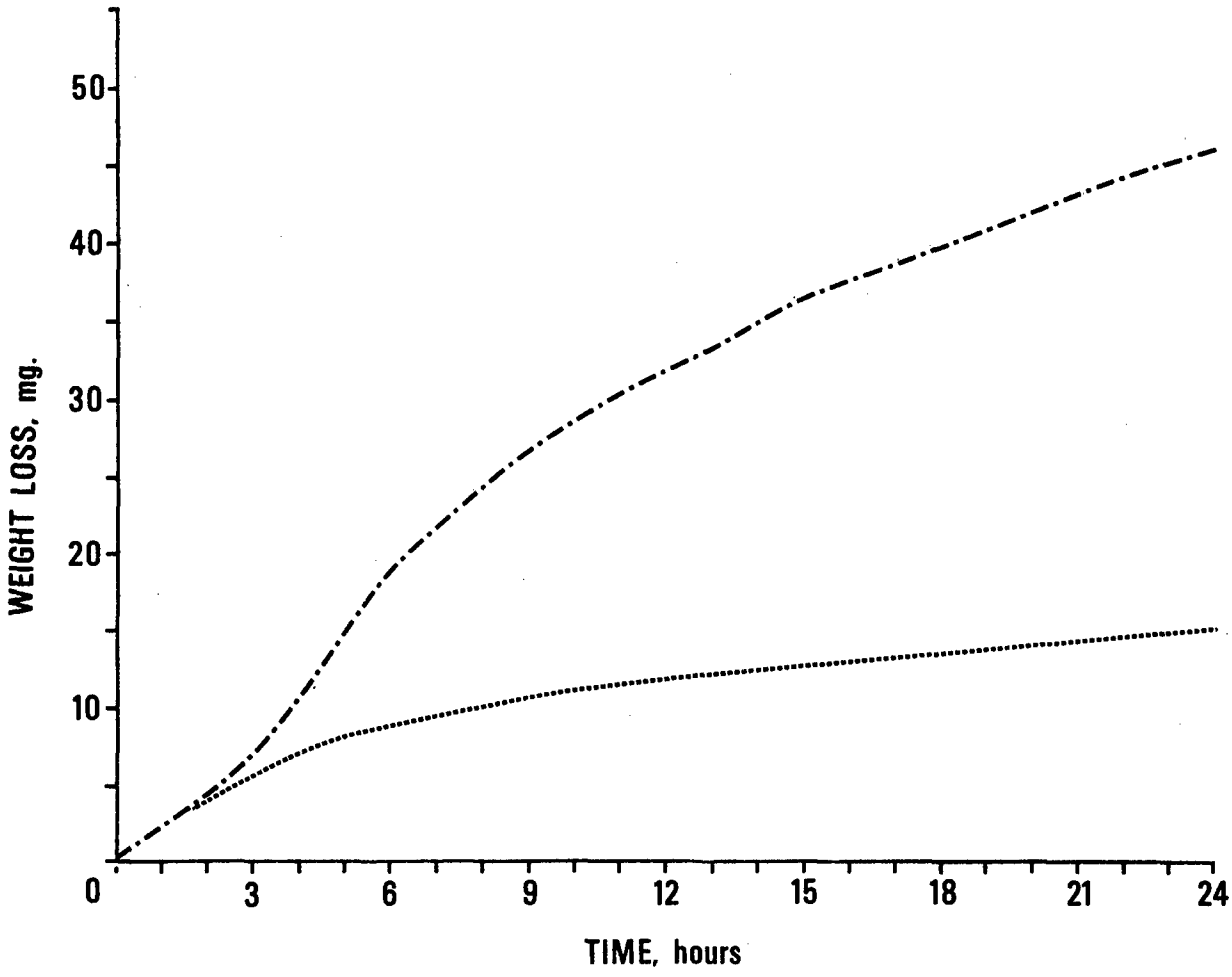

Figure 31. TGA (Upper) and Water Loss (Lower) Curves of SF 565 Silicone Rubber (AF 681).

Specimen Weight - 10.6231 grams

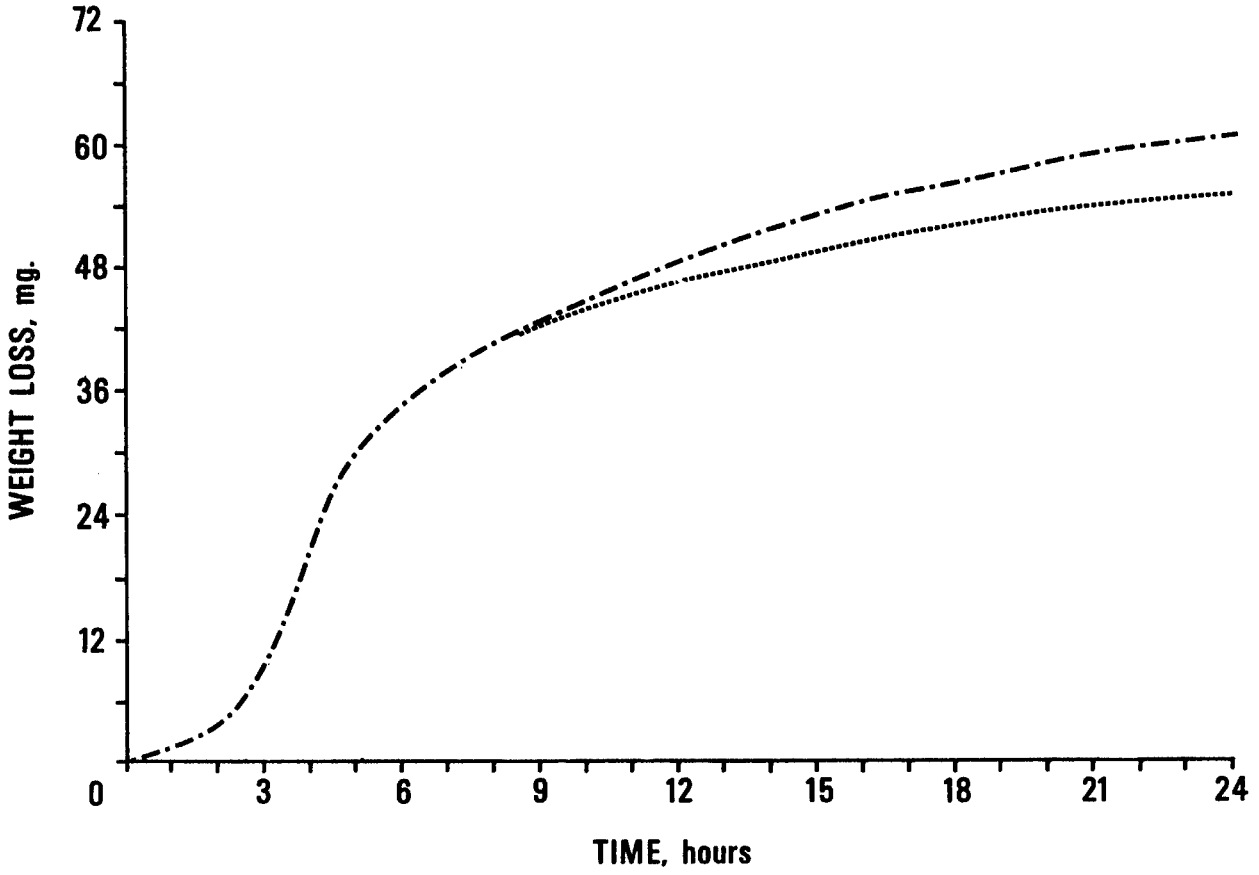

Figure 32. TGA (Upper) and Water Loss (Lower) Curves of Hysol 0151 Sealant (AF 699).

Specimen Weight - 10.9150 grams

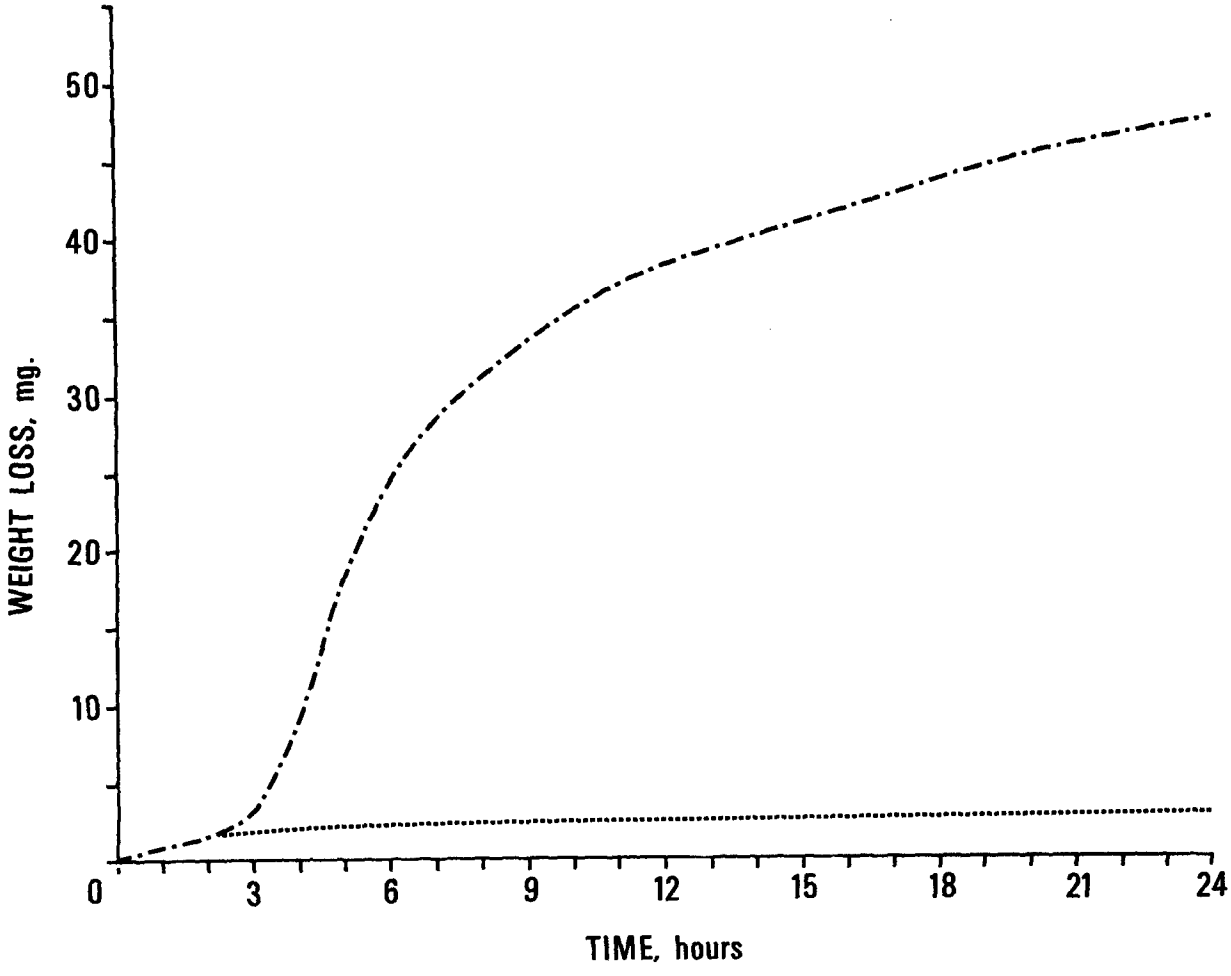

Figure 33. TGA (Upper) and Water Loss (Lower) Curves of Dry Film 350 (AF 711).

Specimen Weight - 1.0900 grams

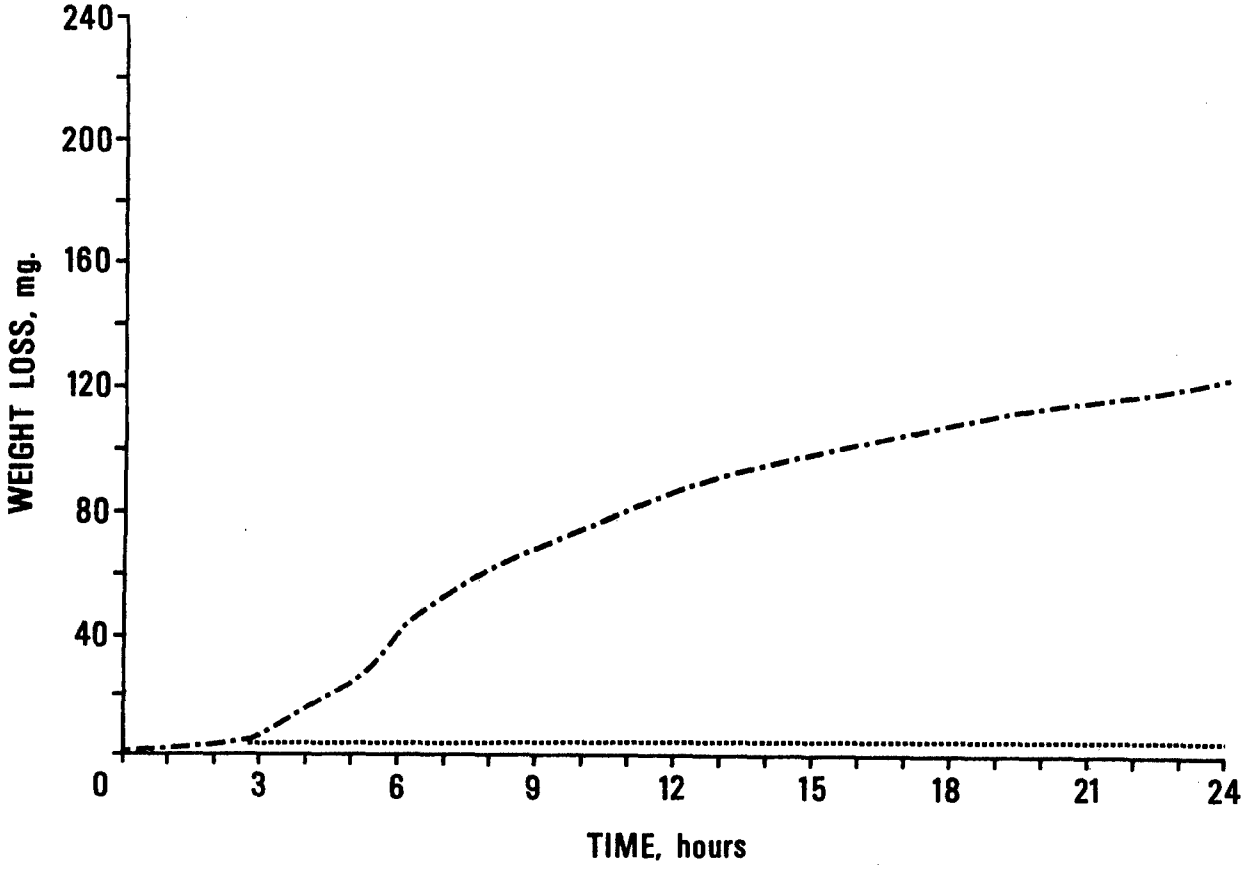

Figure 34. TGA (Upper) and Water Loss (Lower) Curves of N3031A (Lubeco, Inc.) (AF 712).
Specimen Weight - 5.7526 grams

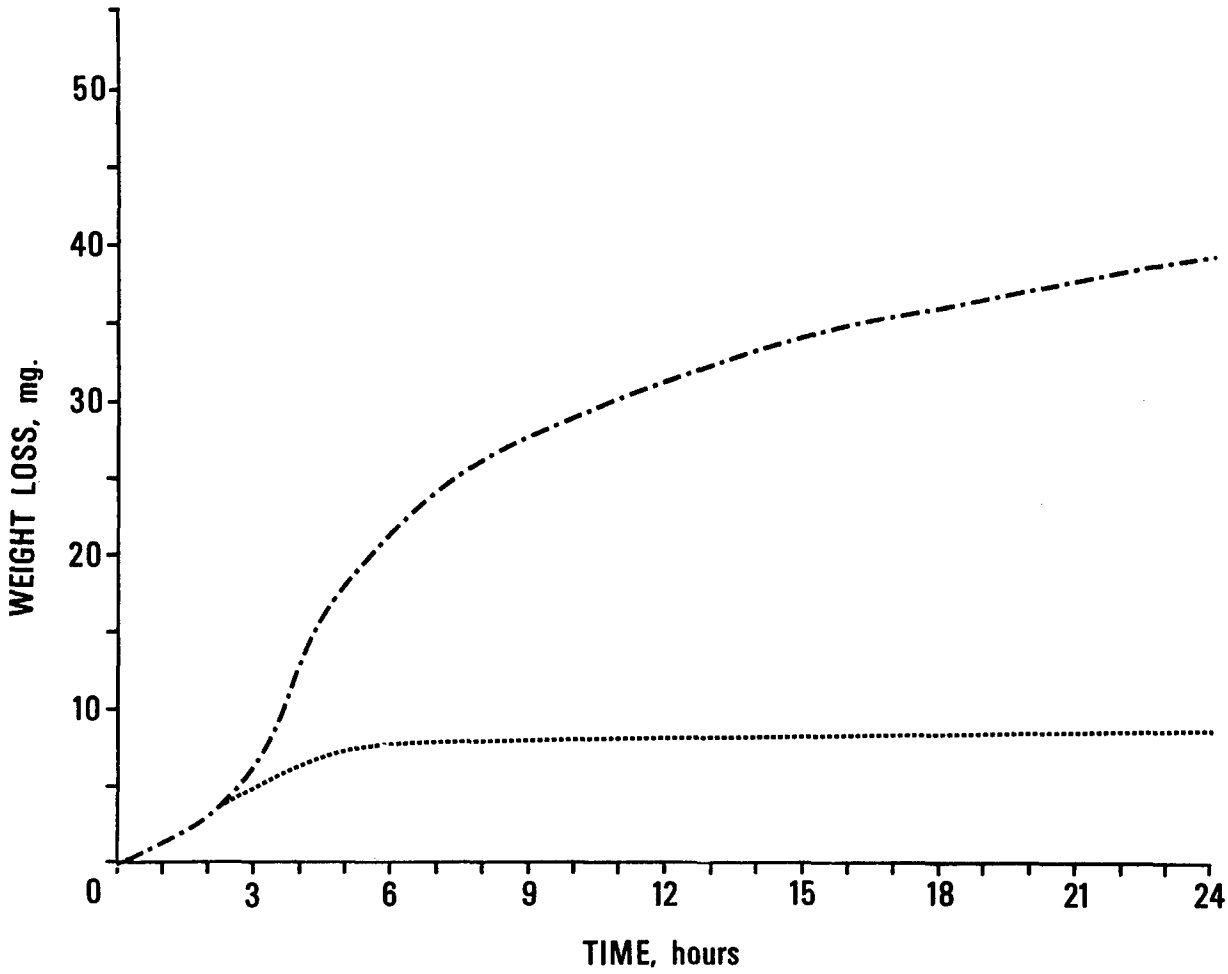

Figure 35. TGA (Upper) and Water Loss (Lower) Curves of S1-350A-Lubricant (AF 713).

Specimen Weight - 1.6112 grams

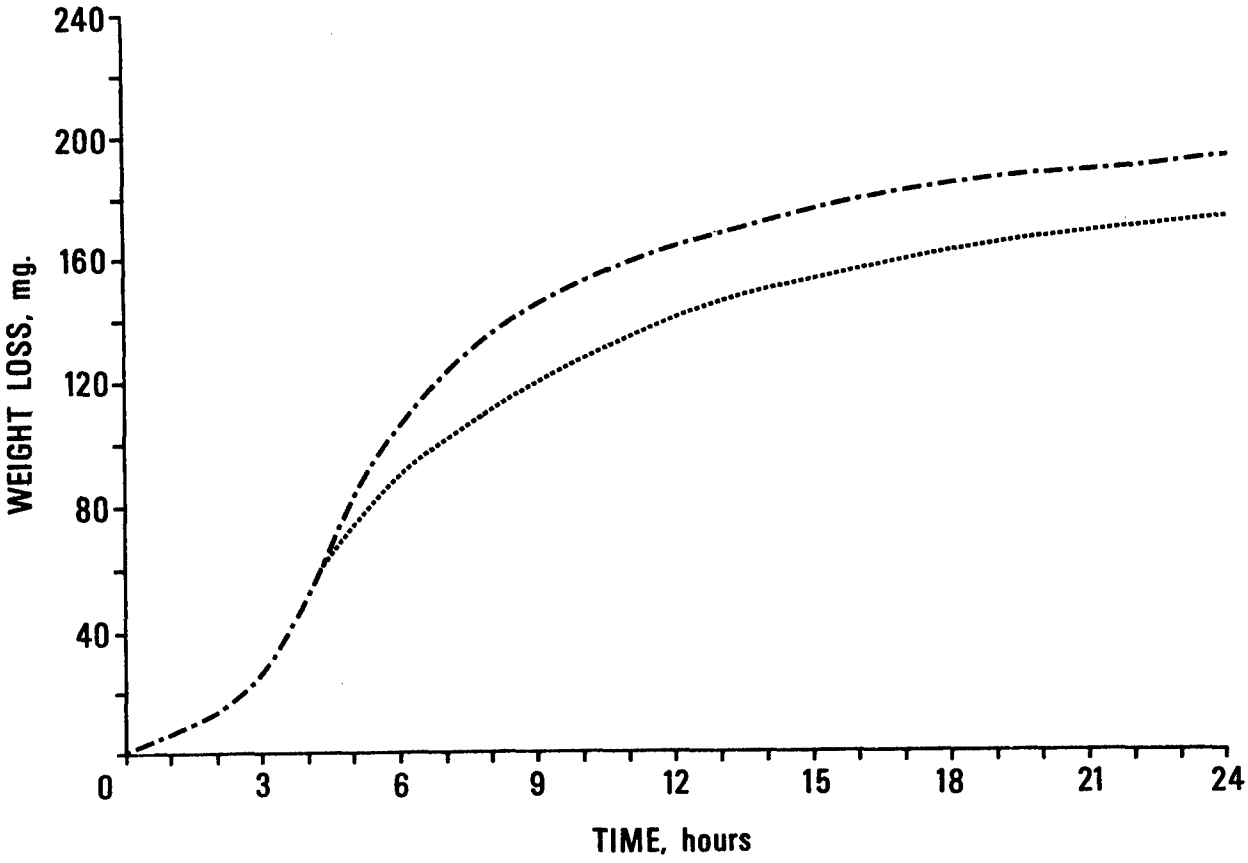

Figure 36. TGA (Upper) and Water Loss (Lower) Curves of 401-F1 Yellow Velvet Paint (AF 718).
Specimen Weight - 9.8587 grams

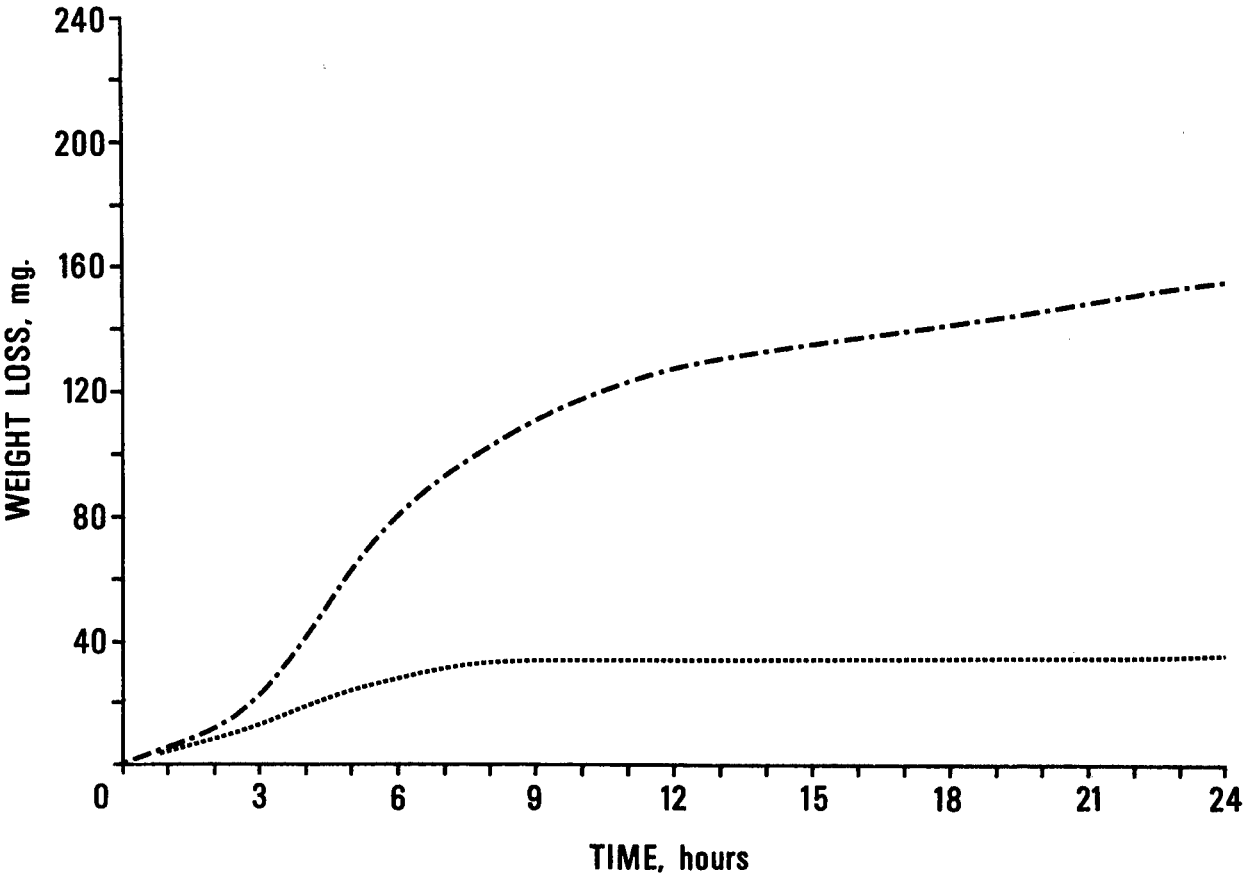

Figure 37. TGA (Upper) and Water Loss (Lower) Curves of 401-H2 Blue Velvet Paint (Dried) (AF 719).
Specimen Weight - 9.2323 grams

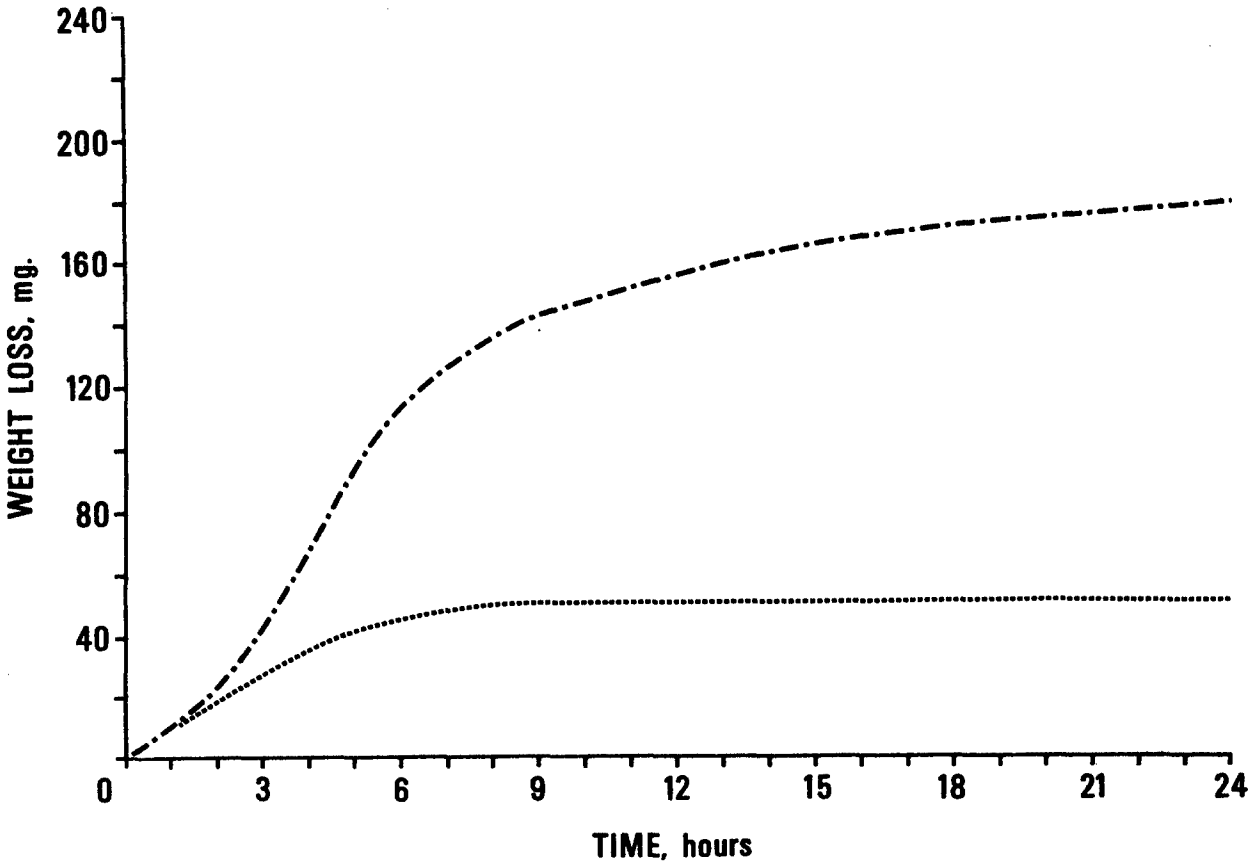

Figure 38. TGA (Upper) and Water Loss (Lower) Curves of 401-H2 Blue Velvet Paint (Not Dried) (AF 719).
Specimen Weight - 7.6986 grams

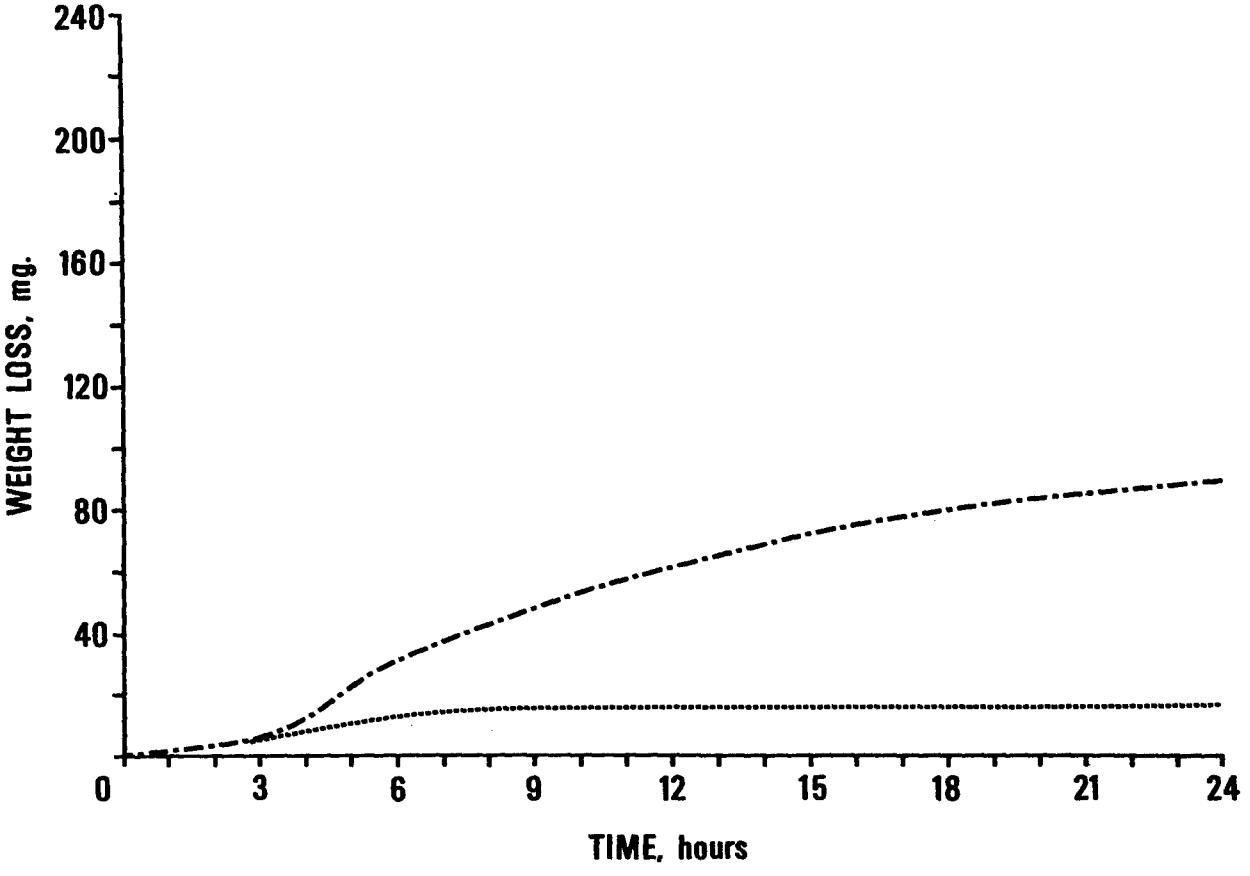

Figure 39. TGA (Upper) and Water Loss (Lower) Curves of 401-J1 Tan Velvet Paint (AF 720).

Specimen Weight - 10.2294 grams

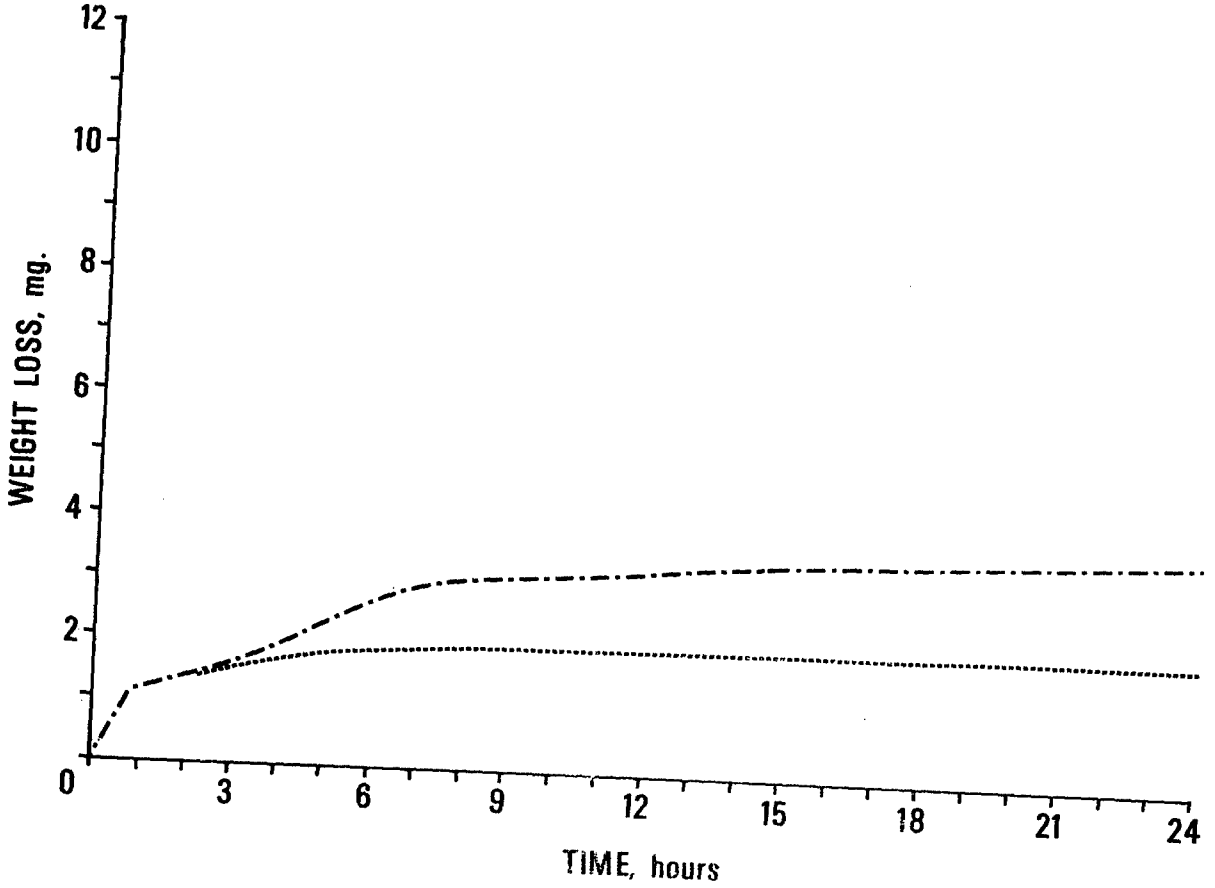

Figure 40. TGA (Upper) and Water Loss (Lower) Curves of 425 Press. Sens. Tape (AF 722).
Specimen Weight - 9.3129 grams

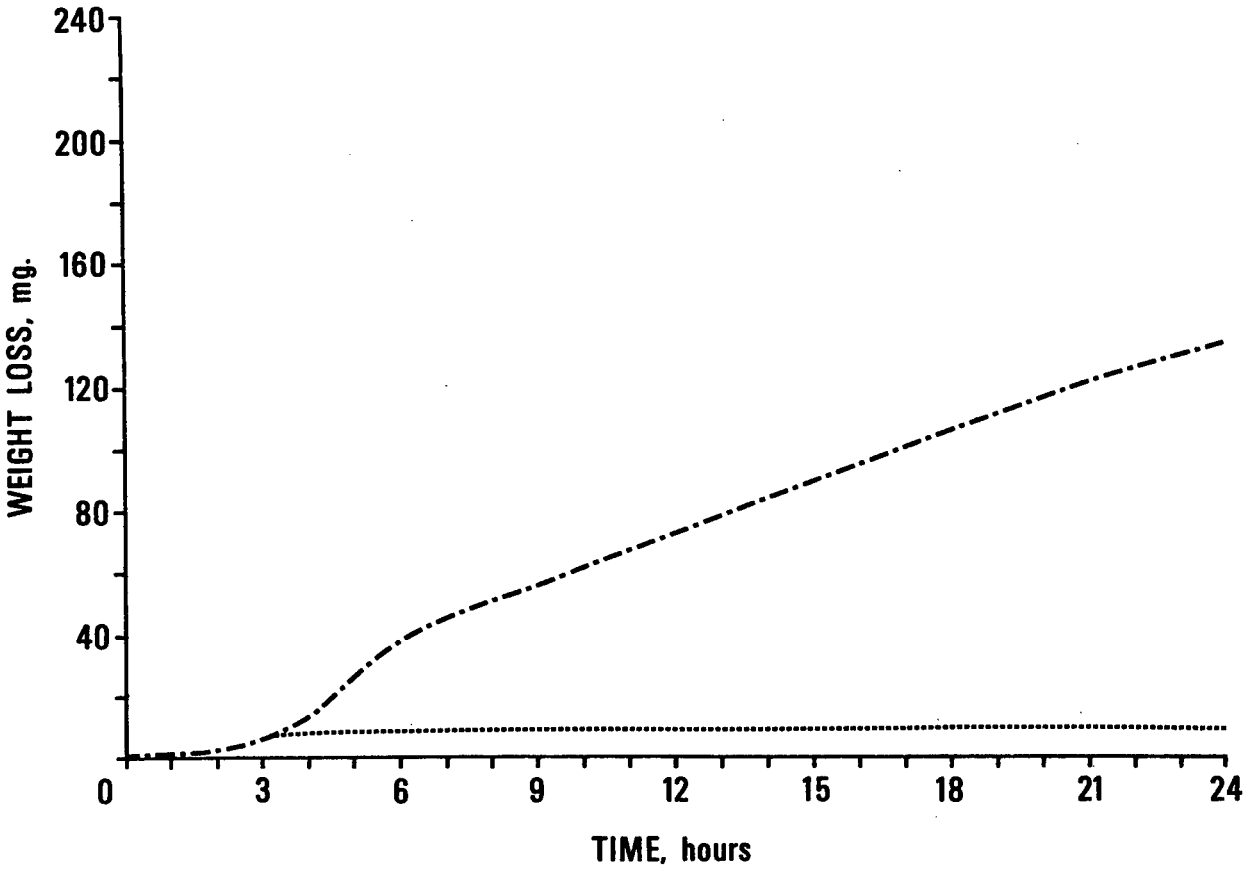

Figure 41. TGA (Upper) and Water Loss (Lower) Curves of EC-880 Adhesive (AF 725).

Specimen Weight - 10.3658 grams

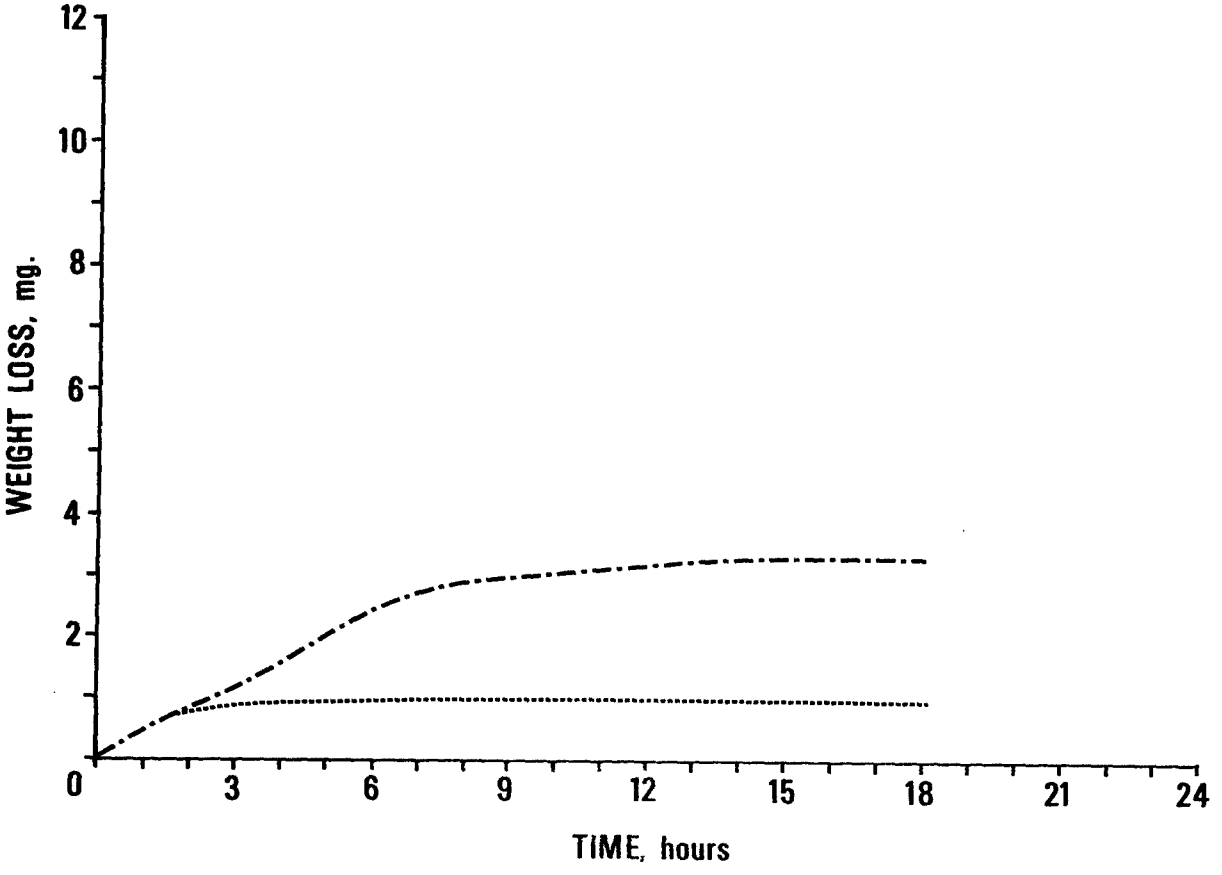

Figure 42. TGA (Upper) and Water Loss (Lower) Curves of Scotchcast 3 (AF 727).

Specimen Weight - 10.5641 grams

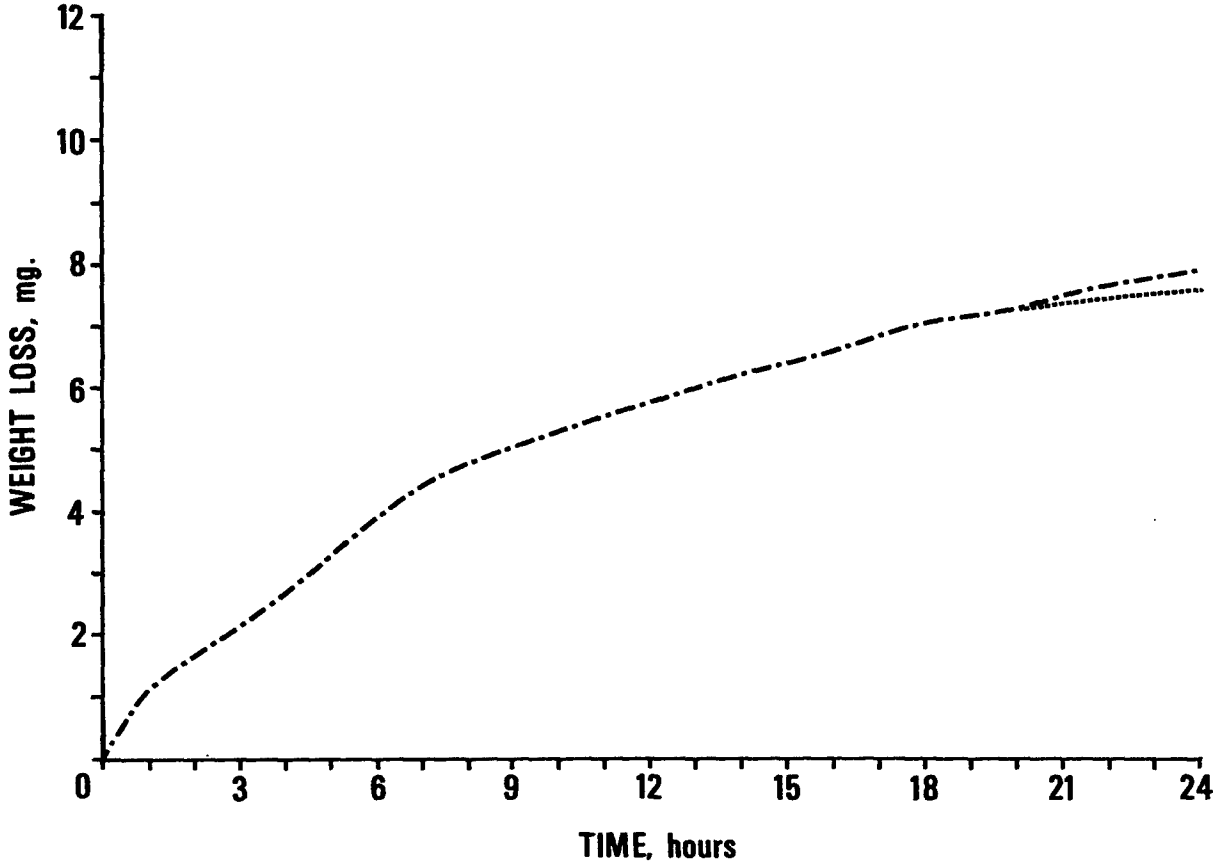

Figure 43. TGA (Upper) and Water Loss (Lower) Curves of Scotchcast 263 (AF 728).

Specimen Weight - 10.0717 grams

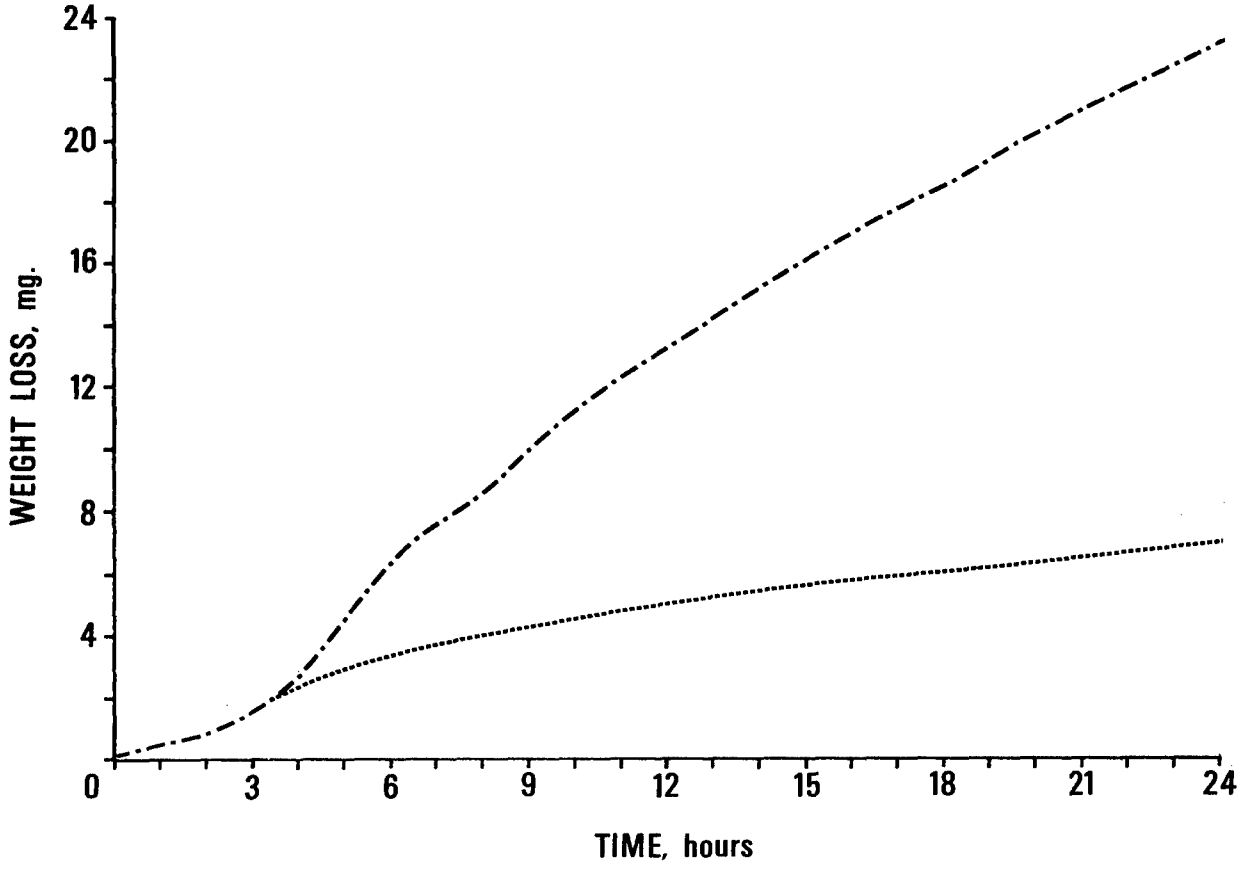

Figure 44. TGA (Upper) and Water Loss (Lower) Curves of Scotchweld 583 (AF 730).

Specimen Weight - 6.6820 grams

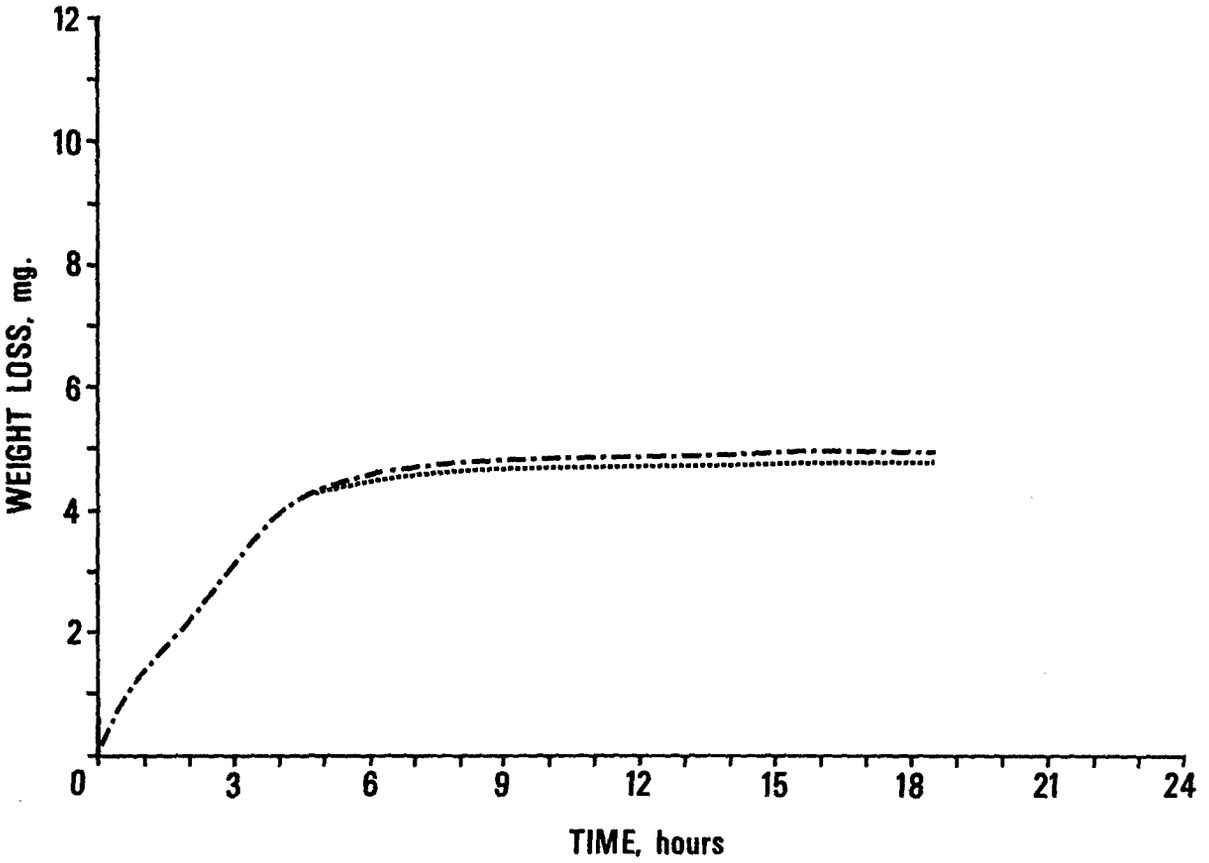

Figure 45. TGA (Upper) and Water Loss (Lower) Curves of E18 Fiberglass Cord (AF 736).

Specimen Weight - 12.1023 grams

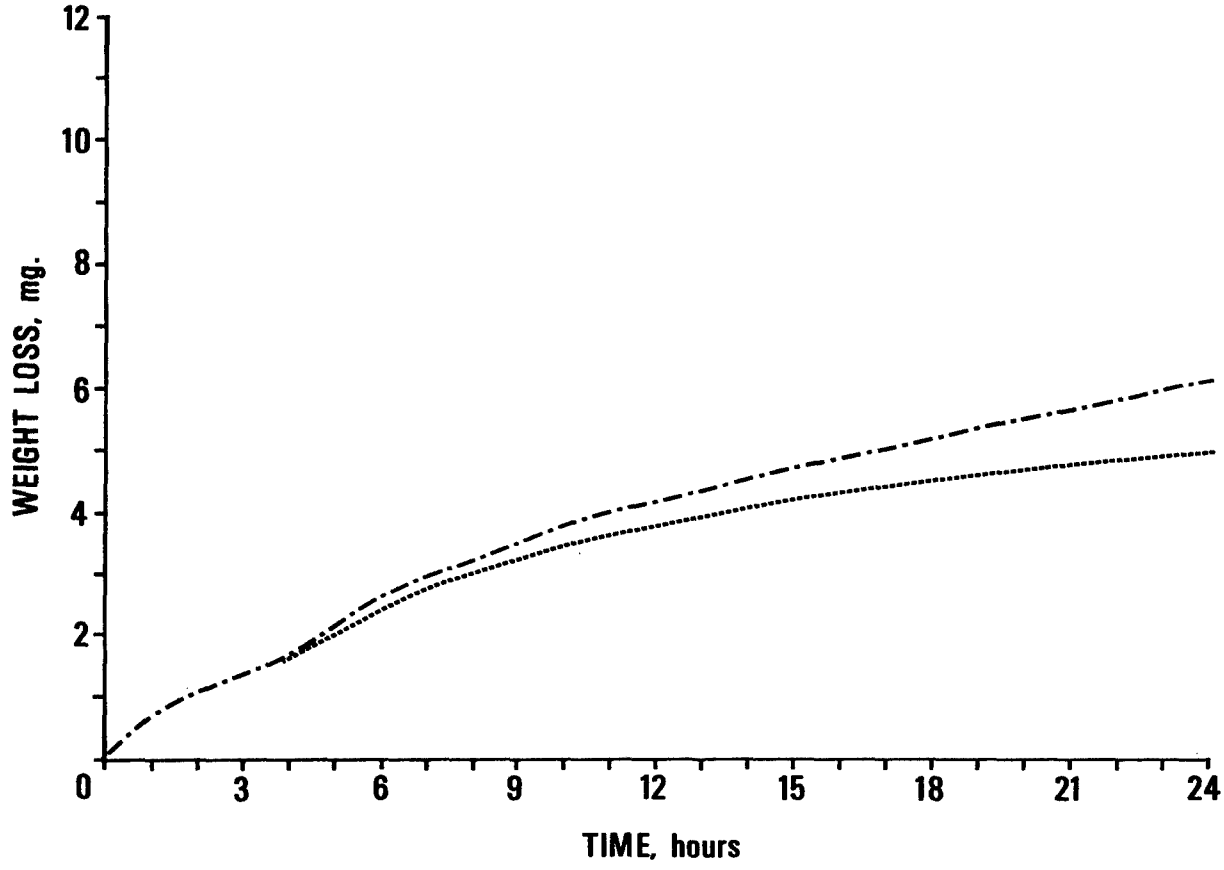

Figure 46. TGA (Upper) and Water Loss (Lower) Curves of EE-6379 Tape (Polyimide Film) (AF 743).
Specimen Weight - 7.8353 grams

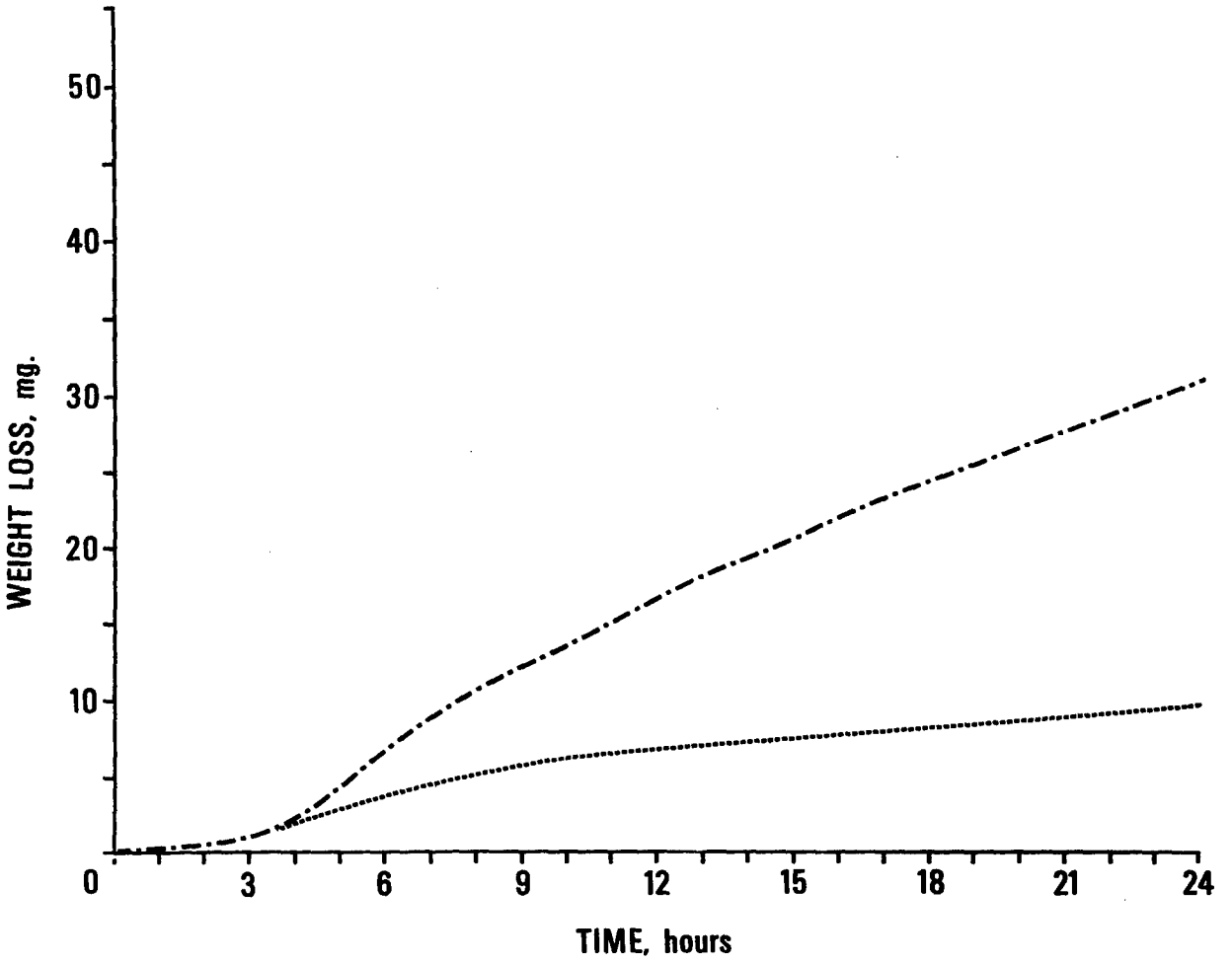

Figure 47. TGA (Upper) and Water Loss (Lower) Curves of EPR Elastomer (AF 749).

Specimen Weight - 9.1003 grams

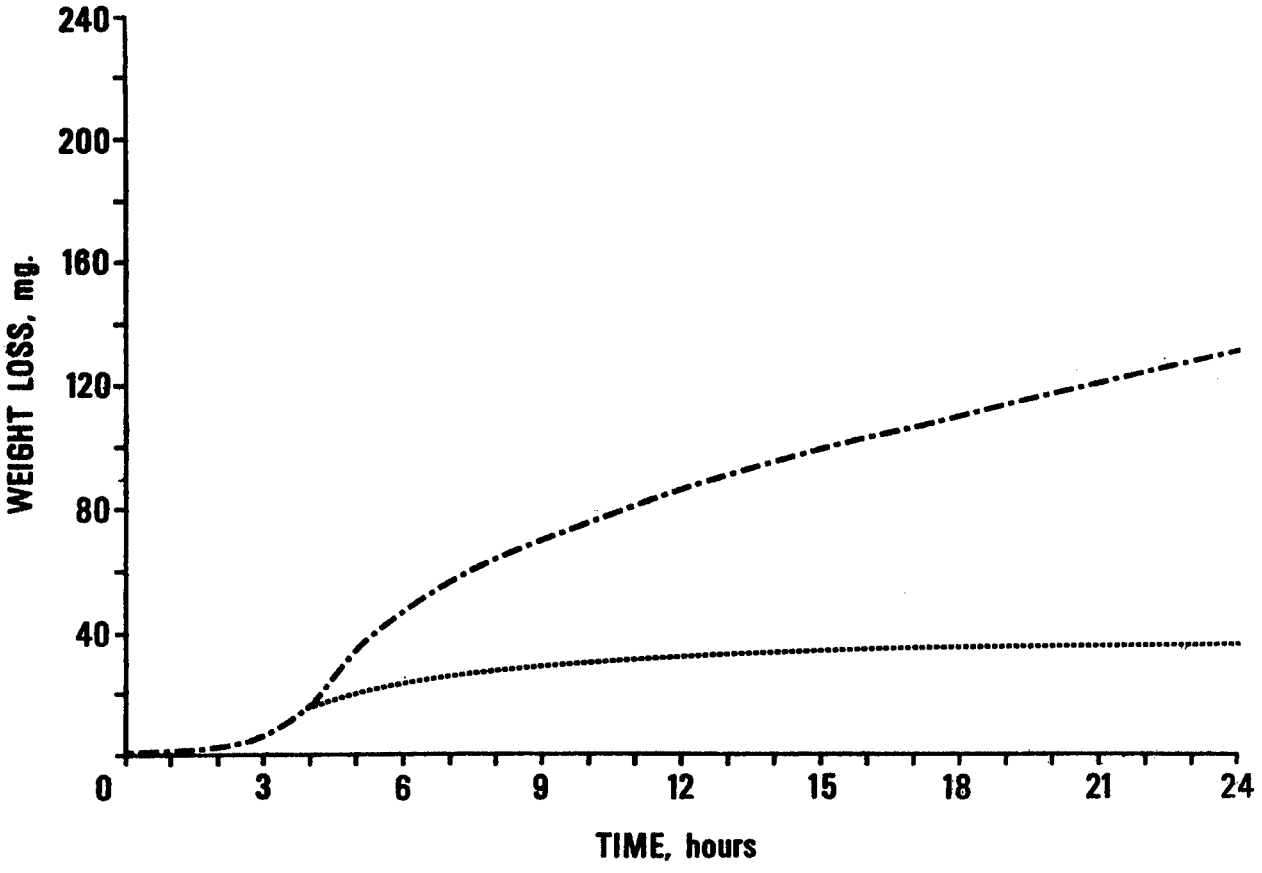

Figure 48. TGA (Upper) and Water Loss (Lower) Curves of F55AP14 Epoxy Enamel (AF 761).

Specimen Weight - 9.8040 grams

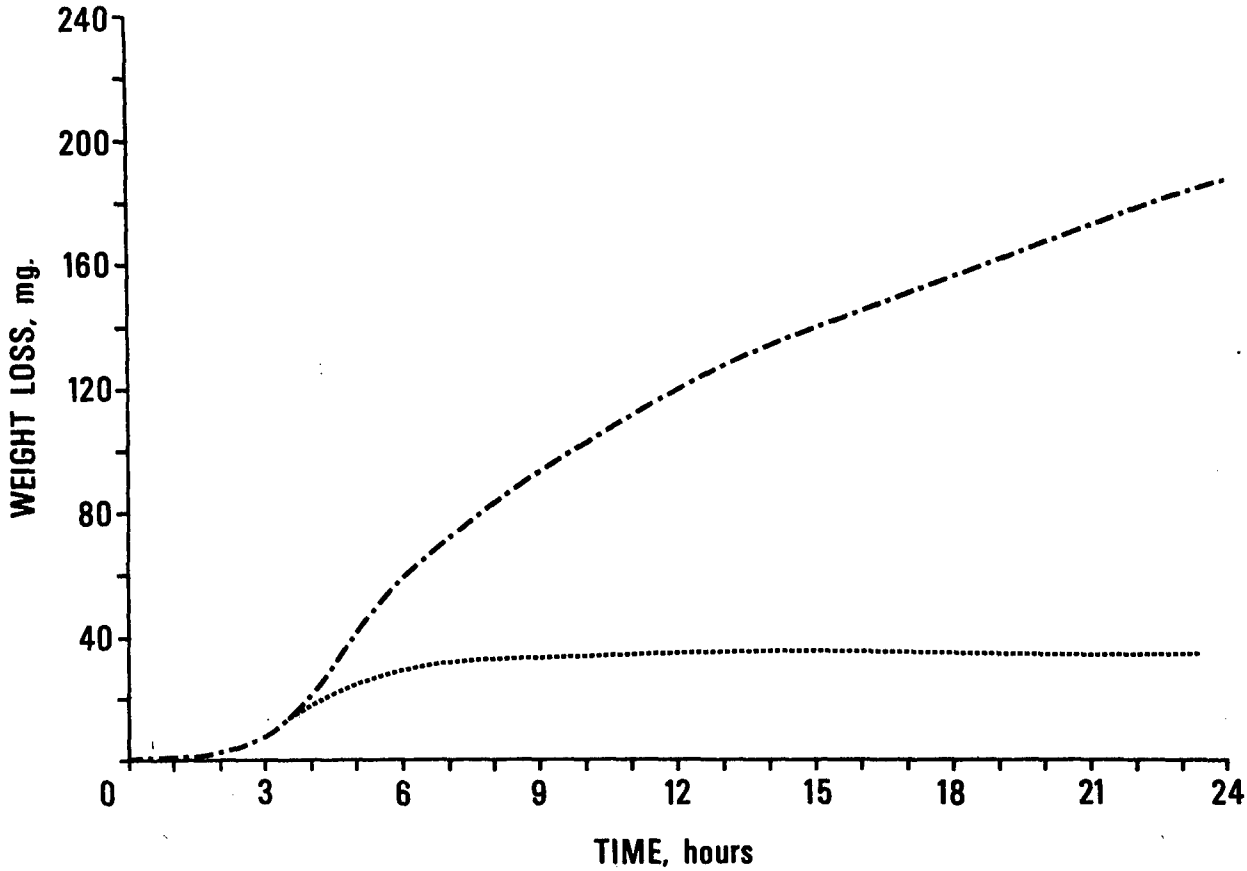

Figure 49. TGA (Upper) and Water Loss (Lower) Curves of F55WP20 Epoxy Paint (AF 764).

Specimen Weight - 9.8569 grams

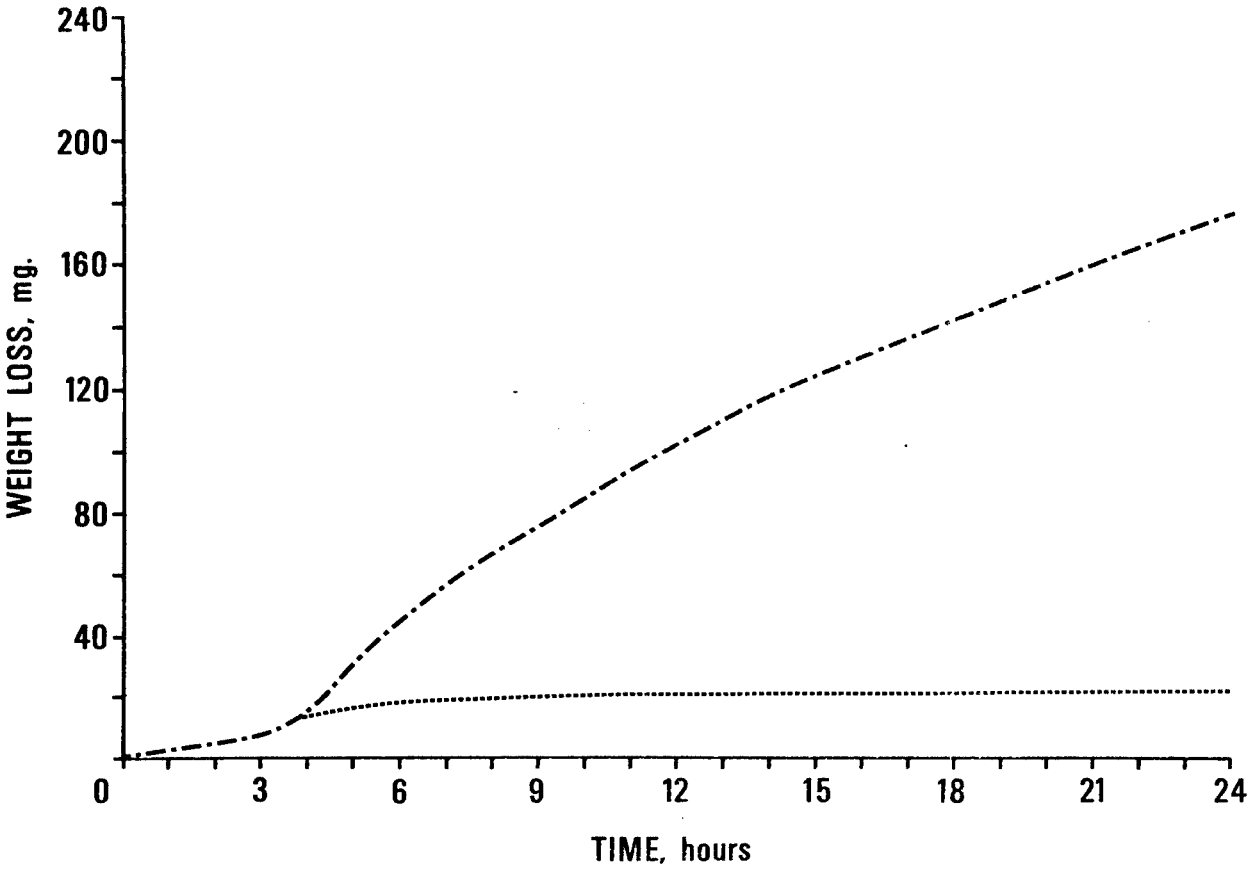

Figure 50. TGA (Upper) and Water Loss (Lower) Curves of U40-VD-5 Paint (AF 766).

Specimen Weight - 6.4900 grams

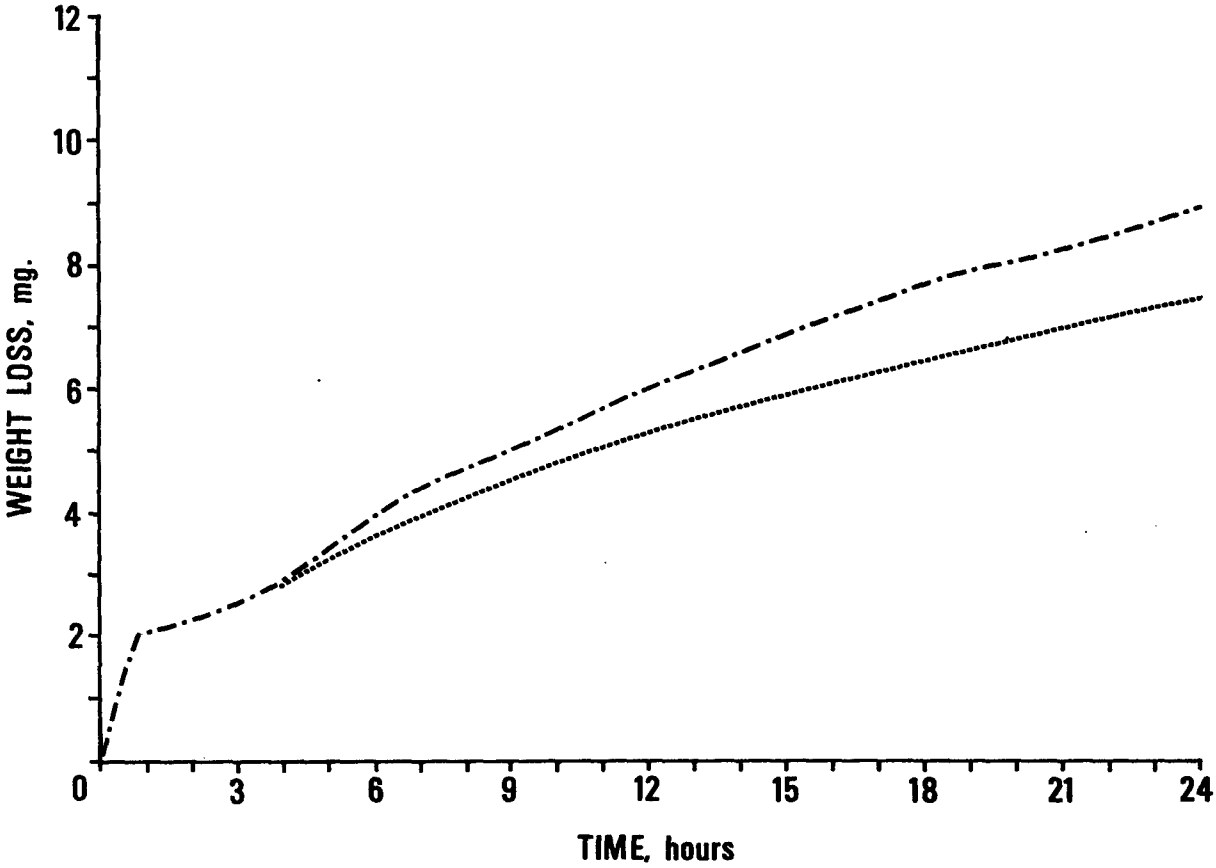

Figure 51. TGA (Upper) and Water Loss (Lower) Curves of Sylgard 182 Potting Compound (AF 786).

Specimen Weight - 11.1972 grams

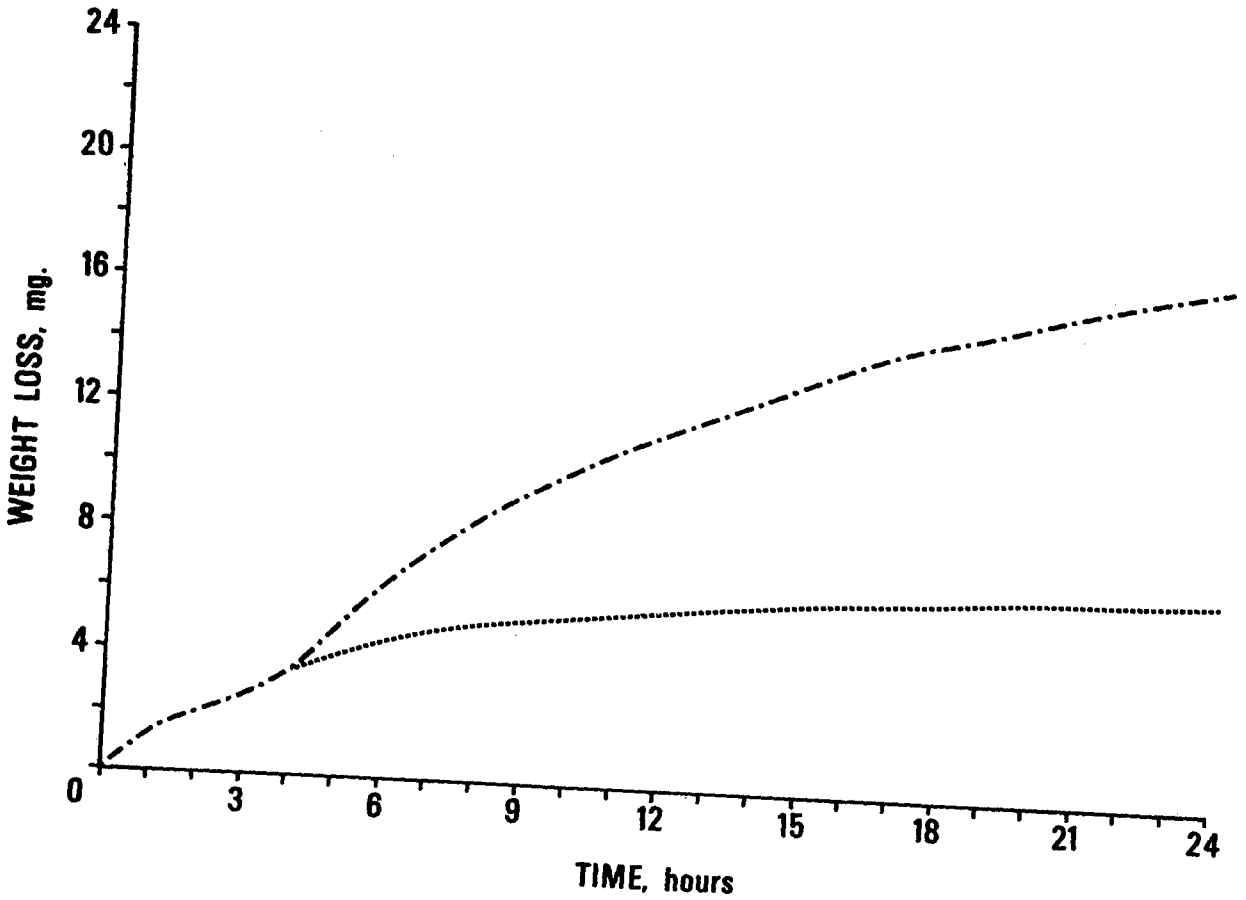

Figure 52. TGA (Upper) and Water Loss (Lower) Curves of DAC-026.

Specimen Weight - 9.8296 grams

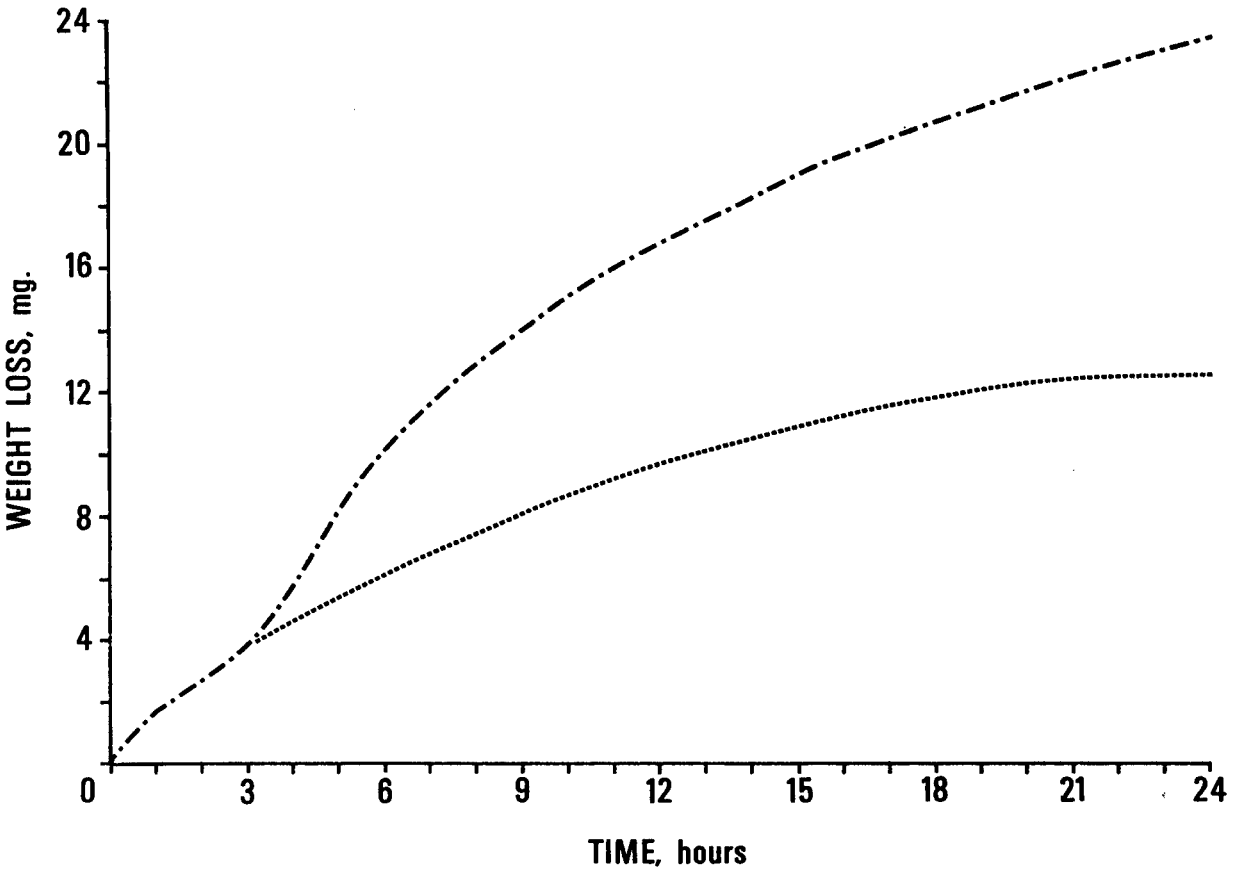

Figure 53. TGA (Upper) and Water Loss (Lower) Curves of DAC-029.

Specimen Weight - 9.4527 grams

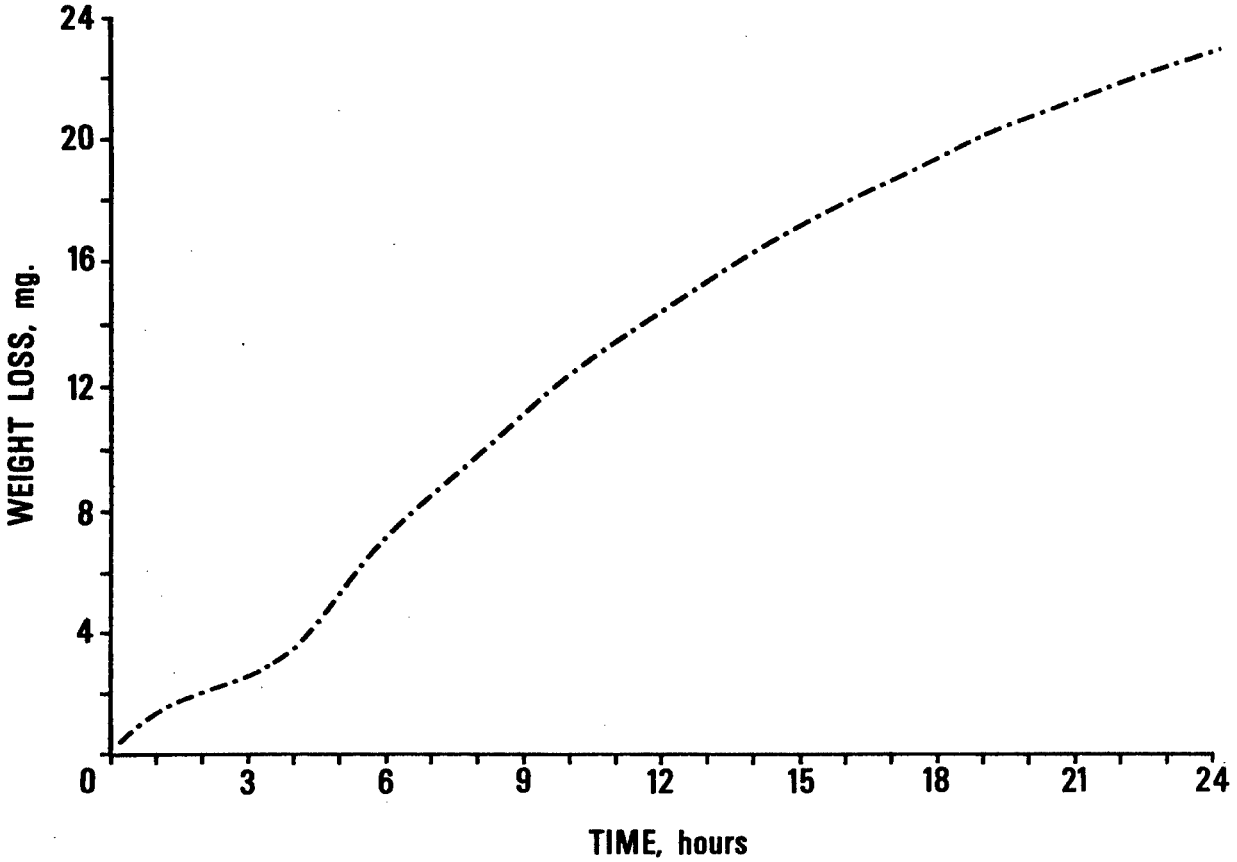

Figure 54. TGA Curve of DAC-030.

Specimen Weight - 10.1023 grams

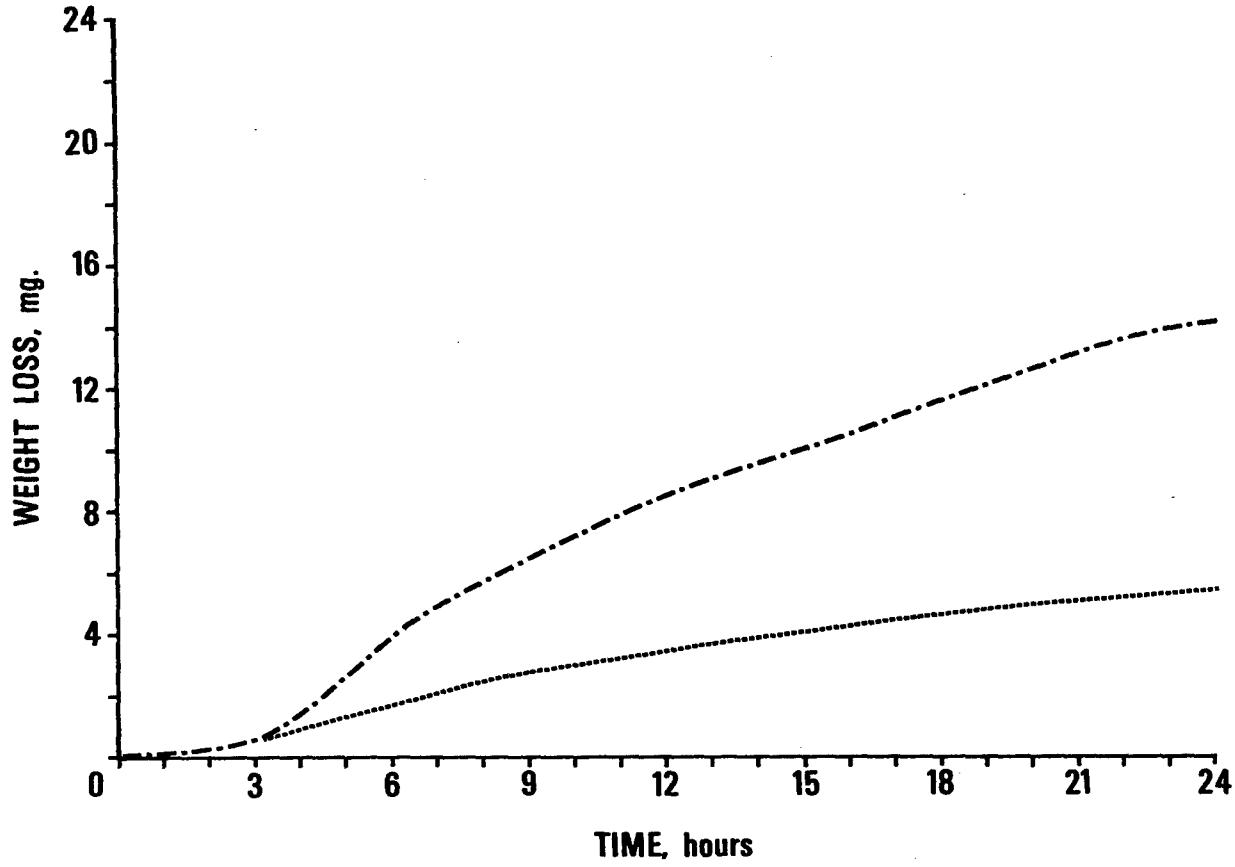

Figure 55. TGA (Upper) and Water Loss (Lower) Curves of DAC-033.

Specimen Weight - 10.7174 grams

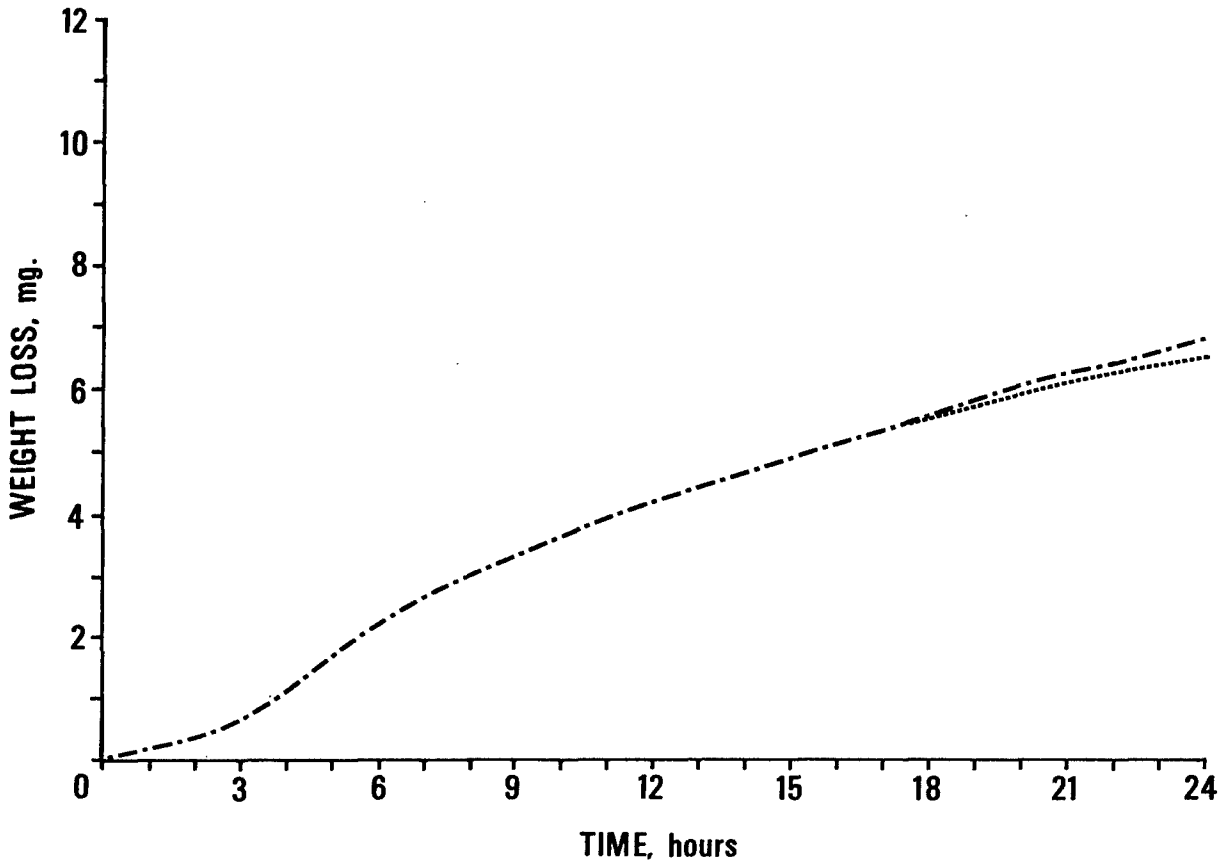

Figure 56. TGA (Upper) and Water Loss (Lower) Curves of DAC-036.

Specimen Weight - 10.6603 grams

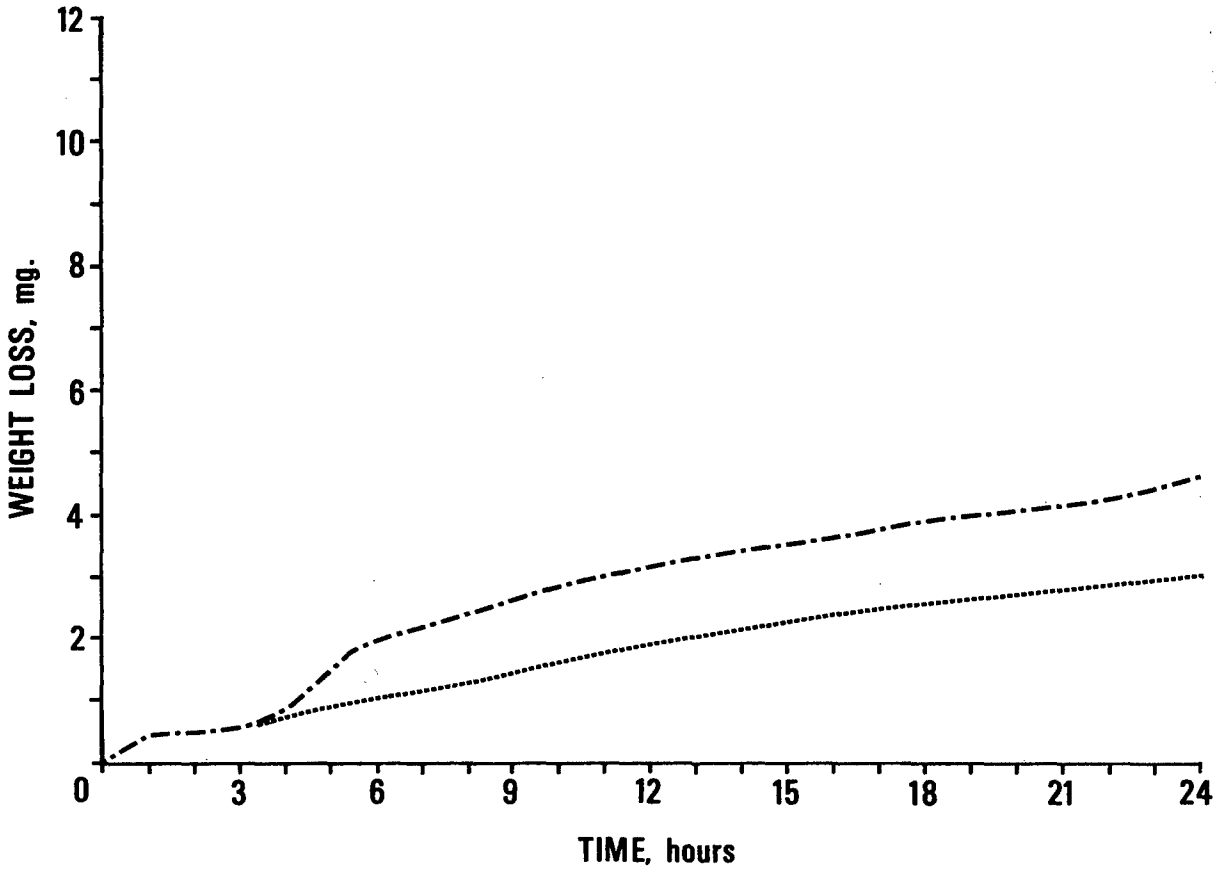

Figure 57. TGA (Upper) and Water Loss (Lower) Curves of DAC-037.

Specimen Weight - 11.1720 grams

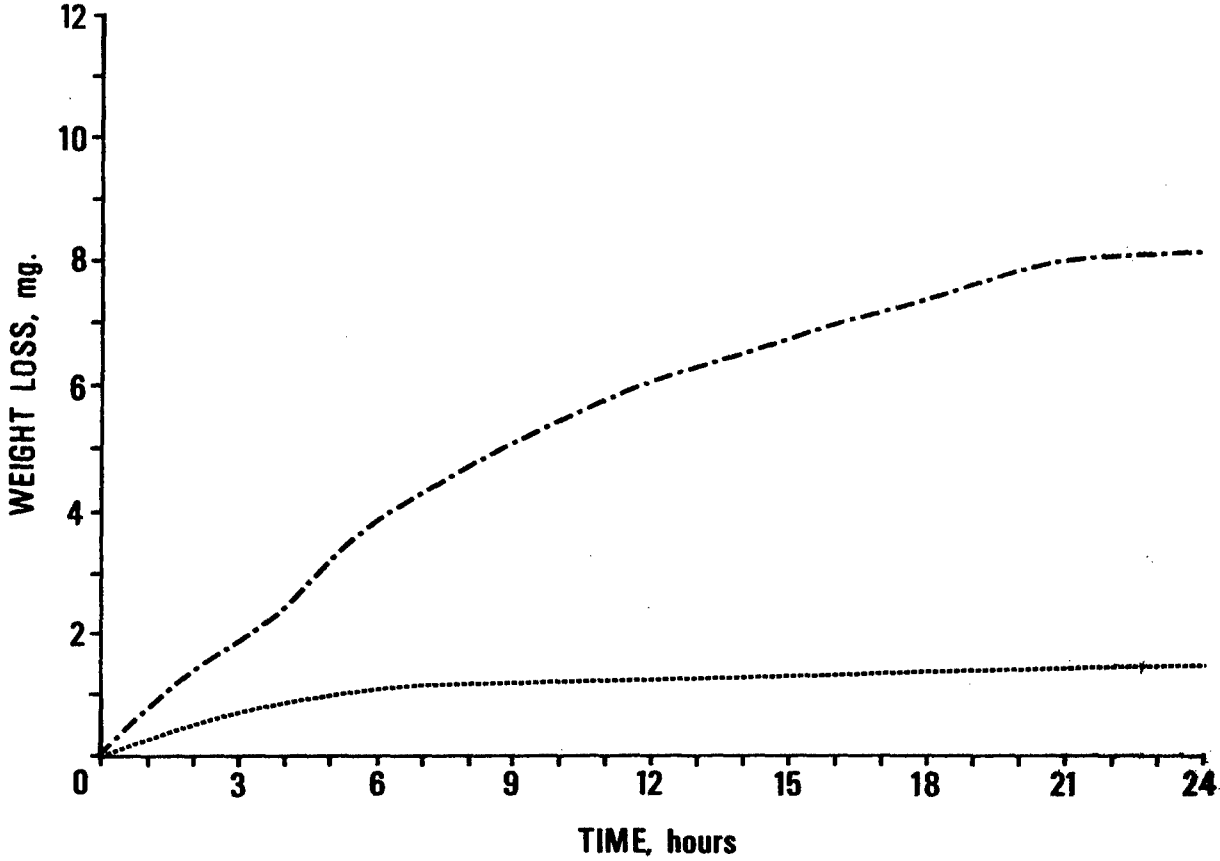

Figure 58. TGA (Upper) and Water Loss (Lower) Curves of DAC-039.

Specimen Weight - 9.8190 grams

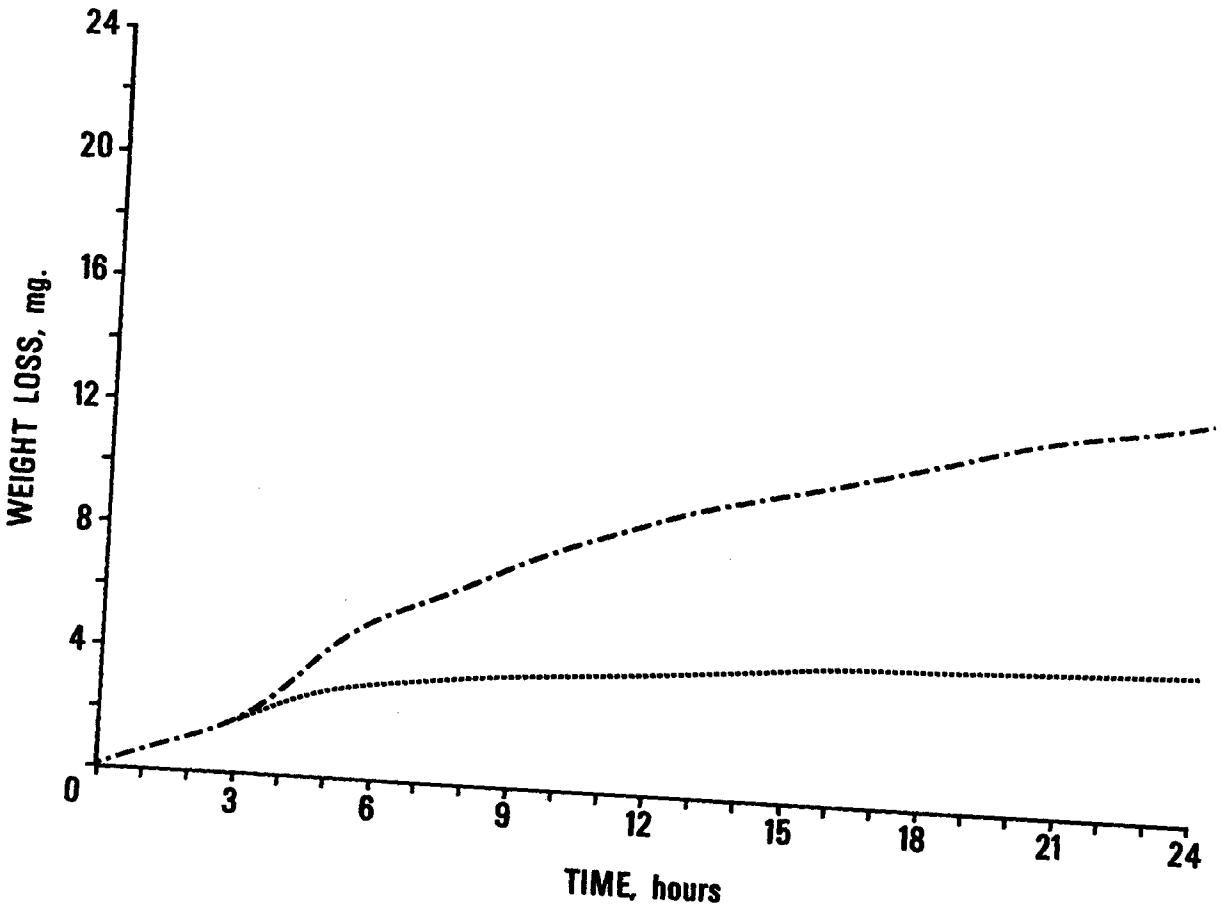

Figure 59. TGA (Upper) and Water Loss (Lower) Curves of DAC-042.

Specimen Weight - 10.2028 grams

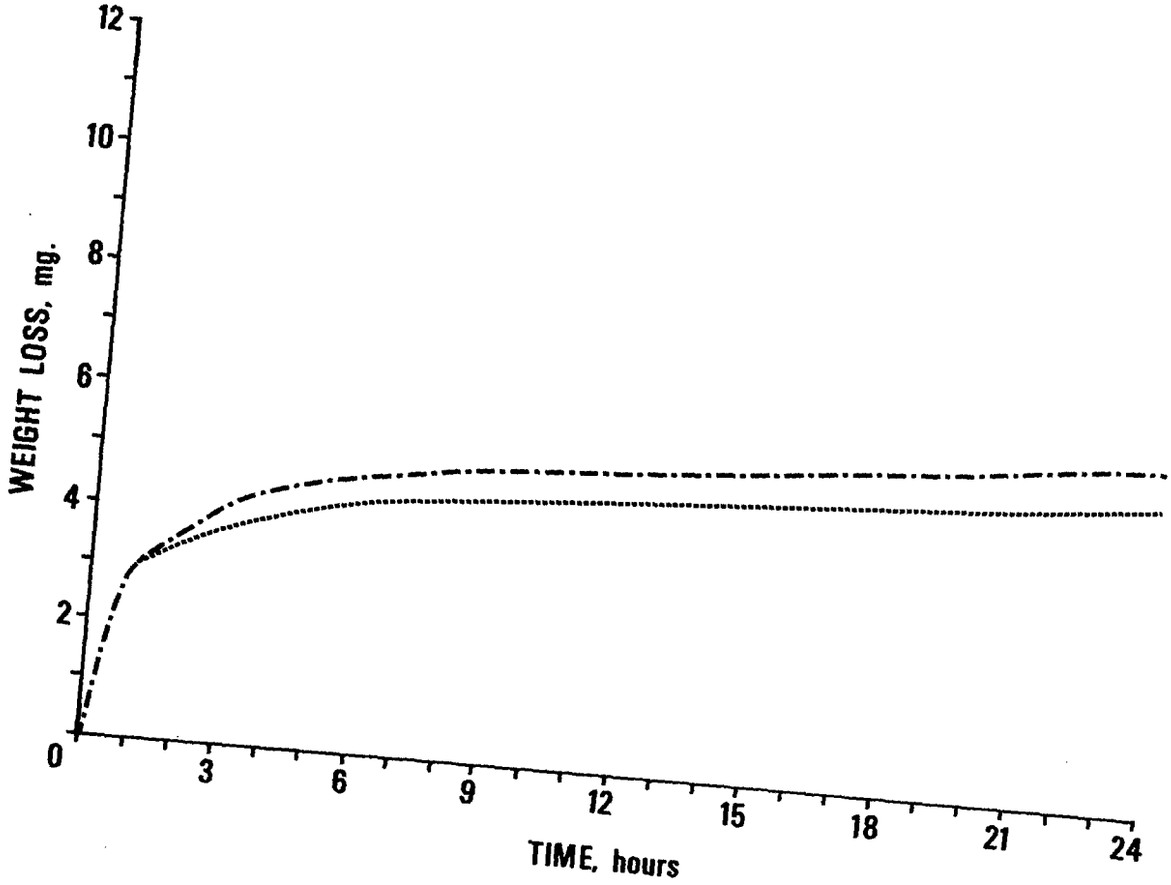

Figure 60. TGA (Upper) and Water Loss (Lower) Curves of DAC-044.
Specimen Weight - 7.9560 grams

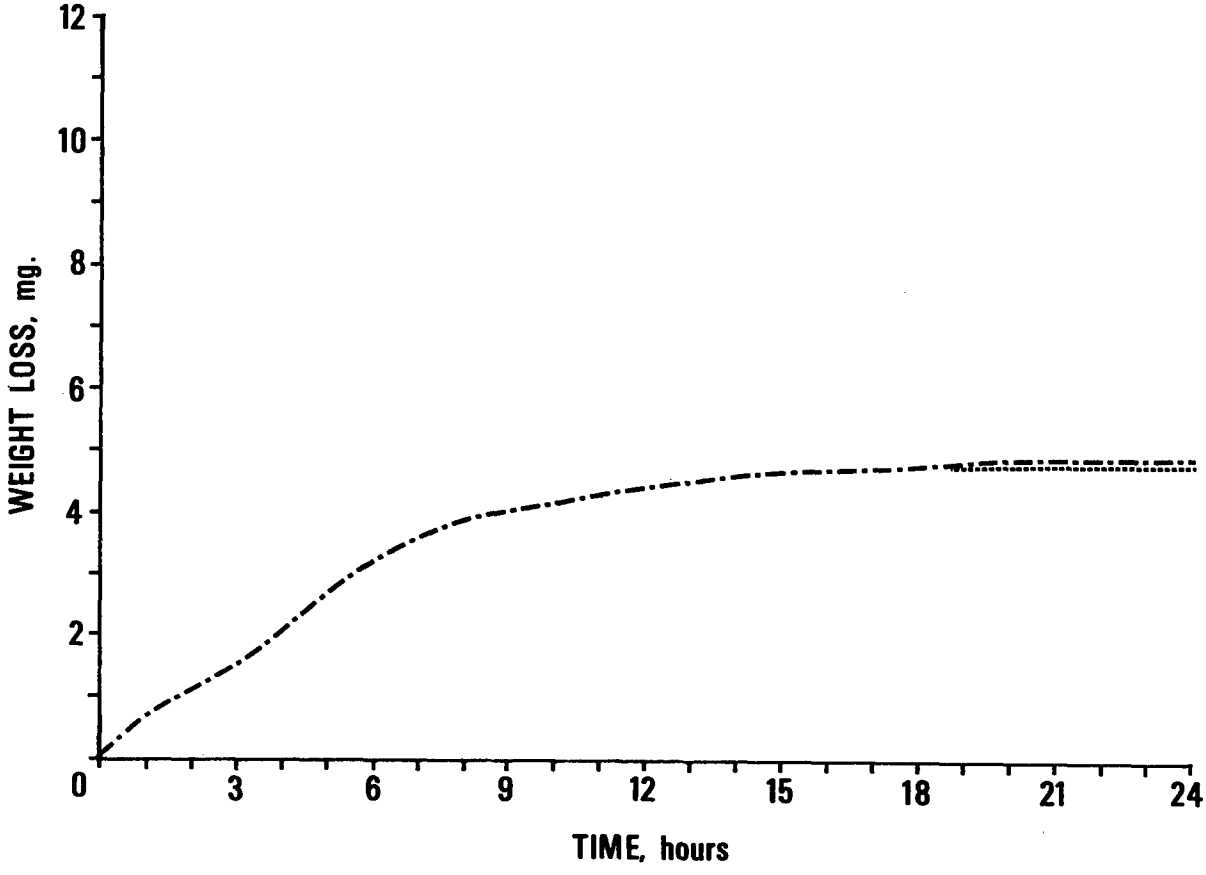

Figure 61. TGA (Upper) and Water Loss (Lower) Curves of DAC-045.

Specimen Weight - 10.1837 grams

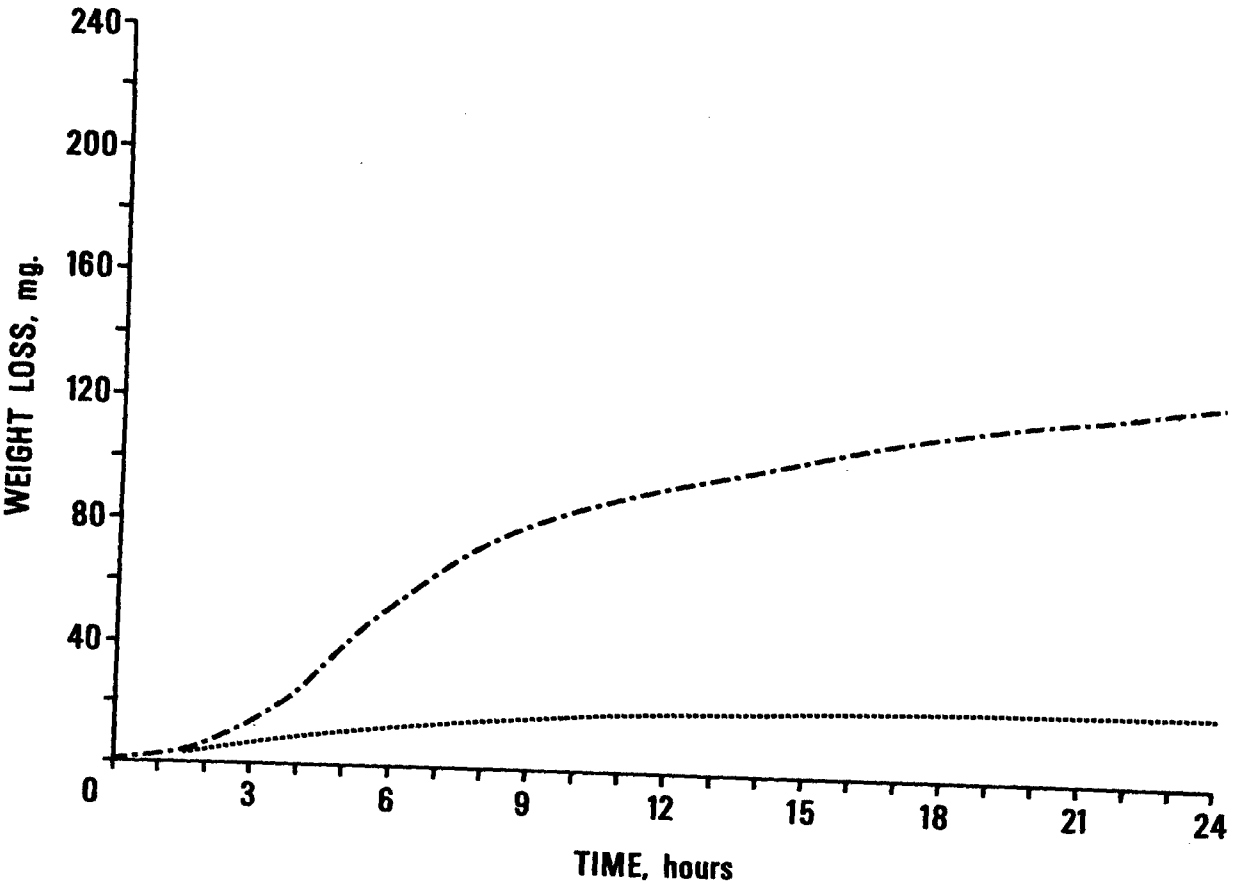

Figure 62. TGA (Upper) and Water Loss (Lower) Curves of DAC-102.

Specimen Weight - 8.1055 grams

APPENDIX II

ANALYTICAL RESULTS

FOR

GAS-OFF EXPERIMENTS

Compounds found as gas-off products from candidate space cabin materials are listed in the following tables. Values for the gas-off product levels are given as: milligrams per 10 grams (mg/10 gms) of the cured candidate material. In some cases, either more or less than 10 grams of material was used, but each yield of gas-off products was normalized to that of a 10-gram sample.

The order of the tables in this appendix is by Air Force serial number. Names of materials are those submitted by the Air Force.

Table IX

GAS-OFF PRODUCTS FROM EPON 828/VERSAMID 140

AF Serial No. 024

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.01	ND	ND
Ethanol	0.09	0.01	0.01
n-Propanol	0.02	ND	ND
4-Methyl-2-pentanone	0.44	ND	ND
Toluene	0.03	ND	ND
Carbon Monoxide	0.06	0.01	0.01

ND - Not detected

Table X

GAS-OFF PRODUCTS FROM POLYESTER GLASS F141

AF Serial No. 129

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.045	0.010	ND
Benzene	0.054	0.069	0.012
C_7 Sat. Hydrocarbon	0.015	0.013	0.024
Toluene	0.42	0.12	0.10
Xylene	0.061	0.019	0.035
C_4 Alkylbenzene	0.071	ND	ND

ND - Not detected

Table XI

GAS-OFF PRODUCTS FROM CORFIL 615 ADHESIVE

AF Serial No. 225

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	0.041	ND	ND
Acetone	0.045	0.037	0.052
Ethanol	0.27	0.17	0.20
Benzene	0.035	ND	ND
Toluene	1.3	1.1	1.2
Xylenes	0.050	0.025	0.030
C_3 Alkylbenzene	0.015	ND	ND
Carbon Monoxide	1.0	0.10	0.22
Methane	0.01	0.001	0.002

ND - Not detected

Table XII

GAS-OFF PRODUCTS FROM VINYLIDENE FLUORIDE, KAYNAR

AF Serial No. 250

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	0.014	ND	ND
2-Butanol	0.010	ND	ND
1,1-Difluoroethane	0.05	ND	ND
SiF_4	0.07	ND	ND
Carbon Monoxide	0.005	0.001	0.001

ND - Not detected

Table XIII

GAS-OFF PRODUCTS FROM PLEX 55 ACRYLIC

AF Serial No. 256

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Benzene	0.022	ND	ND
n-Propanol	0.006	ND	ND
Carbon Monoxide	0.008	0.002	0.003
Methane	0.02	0.01	0.02

ND - Not detected

Table XIV

GAS-OFF PRODUCTS FROM SILICONE ELASTOMER Q2-0078

AF Serial No. 407

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	ND	ND
Ethanol	0.03	0.01	0.02
Toluene	0.01	ND	ND
Carbon Monoxide	0.009	0.001	0.002

ND - Not detected

Table XV

GAS-OFF PRODUCTS FROM EPI REX 510 PAINT

AF Serial No. 410

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methylisobutylketone	2.8	7.7	12.4
Xylenes	0.11	0.029	0.039
Carbon Monoxide	0.005	ND	0.001
Methane	0.008	ND	ND

ND - Not detected

Table XVI

GAS-OFF PRODUCTS FROM DIALLYL PHTHALATE 52

AF Serial No. 417

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C_3 Sat. Hydrocarbon	0.045	ND	ND
Acetone	9.2	0.42	0.029
Methyl-sec-butyl Ether	0.55	ND	ND
t-Butanol	3.7	0.036	0.037
Benzene	0.027	ND	0.004
n-Propanol	0.031	ND	ND
2-Butanol	0.008	ND	ND
Toluene	0.044	0.008	0.013
Xylenes	0.11	ND	ND
Carbon Monoxide	1.0	0.1	0.2
Methane	ND	ND	ND

ND - Not detected

Table XVII

GAS-OFF PRODUCTS FROM HUNISEAL 1B12 LAMINATE

AF Serial No. 465

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.071	0.071	0.11
Methylacetate	0.007	ND	ND
Ethanol	0.006	ND	ND
2-Butanone	0.26	0.026	0.038
Benzene	0.49	0.004	0.011
n-Propanol	0.51	0.004	0.012
4-Methyl-2-pentanone	0.67	0.12	0.26
Toluene	72.0	10.7	15.8
Xylenes	0.15	0.02	0.09
C_3 Alkylbenzene	0.6	0.08	0.13
C_4 Alkylbenzene	0.08	ND	ND
Carbon Monoxide	0.02	0.002	0.005
Methane	0.01	0.005	0.02

ND - Not detected

Table XVIII

GAS-OFF PRODUCTS FROM TAPE-TEMP-R-GLASS

AF Serial No. 491

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	0.004	0.008
Ethanol	0.22	0.03	0.03
n-Propanol	0.03	ND	ND
Carbon Monoxide	0.02	0.001	0.003

ND - Not detected

Table XIX

GAS-OFF PRODUCTS FROM LEXAN 101-01

AF Serial No. 532

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
2-Butanol	0.02	0.010	ND
Toluene	0.22	0.06	0.14
n-Butanol	0.008	0.001	ND
Xylenes	0.07	0.01	0.01
Carbon Monoxide	0.004	0.001	0.001
Chloroethylene	0.05	0.005	0.009

ND - Not detected

Table XX

GAS-OFF PRODUCTS FROM A2 EPOXY ADHESIVE AND ACTIVATOR A

AF Serial No. 619

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Ethanol	0.029	ND	0.004
Carbon Monoxide	0.005	ND	0.001
Methane	0.001	ND	0.001

ND - Not detected

Table XXI

GAS-OFF PRODUCTS FROM
1151 FLEX TUBING (SILICONE RUBBER ON GLASS)

AF Serial No. 623

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.007	ND	ND
Benzene	0.032	ND	ND
Toluene	0.004	ND	ND
Carbon Monoxide	0.002	ND	ND
Methane	0.001	ND	ND

ND - Not detected

Table XXII

GAS-OFF PRODUCTS FROM CHR 3320 SILICONE/GLASS

AF Serial No. 639

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.37	0.12	0.069
Silicone Oil (2 types)	0.090	ND	ND
Benzene	0.078	ND	ND
Xylene	0.030	ND	ND
Carbon Monoxide	0.01	0.01	ND
Methane	0.02	0.02	ND

ND - Not detected

Table XXIII

GAS-OFF PRODUCTS FROM DC-5 LUBE

AF Serial No. 645

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C_4-C_6 Sat. Hydrocarbons	0.052	0.007	0.015
Acetone	0.067	0.007	0.015
Silicone Oil (3 types)	3.8	0.086	0.16
Toluene	1.5	0.053	0.009
Trimethylsilanol	0.30	ND	ND
Xylene	0.10	ND	ND
Carbon Monoxide	0.01	ND	0.005
Methane	0.02	ND	0.02

ND - Not detected

Table XXIV

GAS-OFF PRODUCTS FROM DC-33 LIGHT GREASE

AF Serial No. 646

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.031	0.015	0.019
Silicone Oil (7 types)	4.2	1.2	1.6
Trimethylsilanol	0.082	0.096	0.12
Toluene	0.066	0.043	0.049
Xylenes	0.12	0.03	0.04
Carbon Monoxide	0.01	ND	ND
Methane	0.002	ND	ND

ND - Not detected

Table XXV

GAS-OFF PRODUCTS FROM DC-33 MEDIUM GREASE

AF Serial No. 647

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.072	0.030	0.045
Silicone Oil (6 types)	7.5	0.25	0.34
Trimethylsilanol	0.14	ND	ND
Toluene	0.070	0.14	0.13
Xylenes	0.048	ND	ND
Carbon Monoxide	0.01	0.001	0.003
Methane	0.02	0.005	0.01

ND - Not detected

Table XXVI

GAS-OFF PRODUCTS FROM DC-33 HEAVY GREASE

AF Serial No. 648

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.086	0.045	0.061
Silicone Oil (6 types)	9.8	4.7	4.9
Trimethylsilanol	2.0	0.088	0.080
Toluene	0.080	0.12	0.080
Xylenes	0.074	0.030	0.030
Carbon Monoxide	0.005	ND	ND
Methane	0.002	ND	ND

ND - Not detected

Table XXVII

GAS-OFF PRODUCTS FROM DC-510 LUBE (SILICONE)

AF Serial No. 649

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.015	ND	ND
Silicone Oil (2 types)	0.35	ND	ND
Benzene	0.004	0.015	0.018
Trimethylsilanol	0.044	ND	ND
Toluene	0.46	0.23	0.24
Xylenes	0.01	0.02	0.02
Carbon Monoxide	0.005	0.002	0.003
Methane	0.02	0.005	0.02

ND - Not detected

Table XXVIII

GAS-OFF PRODUCTS FROM RTV 501 POTTING COMPOUND

AF Serial No. 653

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Silicone Oil (2 types)	2.5	0.26	0.43
n-Propanol	1.1	1.8	1.9
2-Butanol	1.2	0.057	0.11
Xylenes	0.15	0.013	ND
Carbon Monoxide	ND	ND	ND
Methane	0.01	ND	ND

ND - Not detected

Table XXIX

GAS-OFF PRODUCTS FROM RTV 732 POTTING COMPOUND

AF Serial No. 655

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Silicone Oil (4 types)	4.0	0.12	0.10
Trimethylsilanol	1.0	0.07	0.005
2,4-Pentanedione	0.5*	ND	ND
Carbon Monoxide	0.004	ND	0.001
Methane	0.05	0.002	0.009

ND - Not detected

*Tentative value, subject to interference from low molecular weight siloxane.

Table XXX

GAS-OFF PRODUCTS FROM SILASTIC 8164

AF Serial No. 658

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.018	-	0.043
Silicone Oil (2 types)	0.28	0.065	0.024
Carbon Monoxide	0.07	ND	0.002
Methane	0.02	0.02	0.03

ND - Not detected

Table XXXI

GAS-OFF PRODUCTS FROM STYCAST 1090/CAT 9

AF Serial No. 669-9

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Benzene	0.016	0.002	0.004
Carbon Monoxide	0.004	ND	0.001
Methane	0.002	ND	ND

ND - Not detected

Table XXXII

GAS-OFF PRODUCTS FROM STYCAST 1090/CAT 11

AF Serial No. 669-11

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Benzene	0.016	0.002	0.005
Carbon Monoxide	0.003	ND	ND
Methane	0.002	ND	ND

ND - Not detected

Table XXXIII

GAS-OFF PRODUCTS FROM STYCAST 2762 POTTING COMPOUND

AF Serial No. 671

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
2-Methyl-1,3-dioxalane	0.27	0.053	0.075
Carbon Monoxide	0.005	0.001	0.001
Methane	0.007	0.01	0.003

Table XXXIV

GAS-OFF PRODUCTS FROM URELANE 5712

AF Serial No. 675

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
sec-Butylamine	0.12	0.082	0.11
Toluene	0.038	0.022	0.031
Xylenes	0.031	0.012	0.017
Carbon Monoxide	0.02	0.008	0.006
Methane	0.01	ND	0.02

ND - Not detected

Table XXXV

GAS-OFF PRODUCTS FROM RTV-511 SILICONE RUBBER

AF Serial No. 678

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C_6 Sat. Hydrocarbons	0.16	0.009	0.004
Ethanol	0.73	0.91	1.3
Silicone Oil	0.28	0.008	0.023
Toluene	0.013	0.004	0.005
Xylene	0.015	0.009	0.012
Carbon Monoxide	0.06	0.003	0.006
Methane	0.02	0.008	0.01

Table XXXVI

GAS-OFF PRODUCTS FROM SE 550 SILICONE ELASTOMER

AF Serial No. 680

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Silicone Oil (2 types)	ND	0.44	4.8
Silicone Oil (3 types)	41.6	ND	ND
Trimethylsilanol	1.0	0.73	0.75
Xylene	0.037	0.002	ND
Carbon Monoxide	0.40	0.005	0.011
Methane	0.08	0.001	0.001

ND - Not detected

Table XXXVII

GAS-OFF PRODUCTS FROM SF 565 SILICONE RUBBER

AF Serial No. 681

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.10	0.022	0.087
Silicone Oil (2 types)	15.2	0.12	1.9
Benzene	1.7	ND	0.023
Toluene	0.006	ND	ND
2,4-Pentanedione	0.16*	ND	ND
Xylenes	0.039	ND	ND
C_3 Alkylbenzenes	0.015	ND	ND
Carbon Monoxide	0.007	ND	ND
Methane	ND	ND	ND

ND - Not detected

*Tentative value, subject to interference from a low molecular weight siloxane.

Table XXXVIII

GAS-OFF PRODUCTS FROM HYSOL 0151 SEALANT

AF Serial No. 699

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	0.02	ND	0.01
Ethanol	0.007	ND	ND
Benzene	0.26	ND	0.006
n-Propanol	0.007	ND	ND
2-Butanol	0.10	ND	ND
Toluene	0.19	ND	0.004
n-Butanol	0.50	0.04	ND
Methylethylbenzene	0.01	ND	ND
Dichlorodifluoromethane	0.3	0.002	0.003
Carbon Monoxide	0.13	0.002	0.002

ND - Not detected

Table XXXIX

GAS-OFF PRODUCTS FROM 401-F1 YELLOW VELVET PAINT

AF Serial No. 718

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
n-Propanol	0.017	0.013	0.008
n-Butanol	0.038	0.007	0.015
Xylenes	0.49	0.19	0.13
2-Ethoxyethanol	0.400	0.13	0.087
2-Ethoxyethylacetate	2.0	0.40	0.34
2-(2-Ethoxyethoxy)-ethylacetate	0.019	ND	ND
Carbon Monoxide	0.03	ND	0.002
Methane	0.02	ND	0.004

ND - Not detected

Table XL

GAS-OFF PRODUCTS FROM EC-880 ADHESIVE

AF Serial No. 725

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Pentane	0.06	0.003	0.007
Methylpentane } Hexane	2.8	1.6	2.5
Methylpentene } Dimethylpentane	15.6	10.0	14.8
Hexene	0.94	2.0	5.8
Heptane } Methylhexene	37.1	14.8	22.6
Dimethylpentene	14.3	5.4	9.4
Acetone	91.2	32.2	52.0
C_8 Saturated Hydrocarbons	3.1	0.92	1.3
2-Butanone	53.5	5.6	7.1
Benzene	0.37	0.046	0.095
C_8 Unsaturated Hydrocarbons	0.17	0.025	0.050
C_9 Saturated & Unsaturated Hydrocarbons	0.10	0.012	0.012
Toluene	3.2	1.1	1.8
C_{10} Saturated & Unsaturated Hydrocarbons	0.029	ND	ND
C_{11} Saturated & Unsaturated Hydrocarbons	0.007	ND	ND
Xylenes	0.20	0.042	0.05
Cyclohexanone	0.18	0.036	0.03
C_{11}-C_{15} Saturated & Unsaturated Hydrocarbons	0.26	ND	ND
Methane	0.03	ND	ND
Carbon Monoxide	0.6	0.005	0.009

ND - Not detected

Table XLI

GAS-OFF PRODUCTS FROM SCOTCHCAST 3

AF Serial No. 727

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Silicone Oil	0.22	ND	ND
Ethanol	0.05	0.01	0.03
Acetone	0.24	ND	ND
C_7 Unsaturated Hydrocarbons	0.20	ND	ND
C_8 Saturated Hydrocarbons	0.038	ND	ND
C_8 Unsaturated Hydrocarbons	0.069	ND	ND
C_9 Unsaturated Hydrocarbons	0.19	ND	ND
Hexanone	0.13	ND	ND
Toluene	0.17	ND	ND
C_{10}-C_{11} Unsaturated Hydrocarbons	0.15	ND	ND
Xylenes	0.39	ND	ND
C_{11}-C_{14} Saturated & Unsaturated Hydrocarbons	0.09	ND	ND
Methane	0.02	ND	ND
Carbon Monoxide	0.23	0.002	0.005

ND - Not detected

Table XLII

GAS-OFF PRODUCTS FROM SCOTCHCAST 263

AF Serial No. 728

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Benzene	0.016	0.001	0.004
Carbon Monoxide	0.004	0.001	0.002
Methane	0.001	ND	ND

ND - Not detected

Table XLIII

GAS-OFF PRODUCTS FROM SCOTCHWELD 583

AF Serial No. 730

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Ethylene	0.54	ND	ND
C_3-C_5 Mono-olefin	0.033	ND	ND
Acetone	0.35	0.065	0.087
C_6 Mono-olefin	0.11	ND	ND
Ethanol	6.6	1.9	1.8
2-Butanone	0.58	0.11	0.10
C_7 Mono-olefin	0.13	ND	ND
n-Butanol	0.11	0.006	0.024
C_8-C_{14} Mono-olefin	7.0	ND	ND
Butylbenzene	0.15	ND	ND
Carbon Monoxide	0.40	0.05	0.08
Methane	0.02	0.001	0.003

ND - Not detected

Table XLIV

GAS-OFF PRODUCTS FROM EE-6379 TAPE (POLYIMIDE FILM)

AF Serial No. 743

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.021	0.001	0.001
2-Propanol	0.33	0.088	0.22
Silicone Oil	0.026	0.003	ND
Benzene	0.055	ND	ND
n-Propanol	0.016	0.010	0.015
Trimethylsilanol	0.98	0.029	0.023
Xylene	0.18	ND	ND
Carbon Monoxide	ND	0.005	0.009
Methane	0.02	0.008	0.02

ND - Not detected

Table XLV

GAS-OFF PRODUCTS FROM EPR ELASTOMER

AF Serial No. 749

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Butane	0.13	ND	ND
Acetone	0.023	ND	ND
C_6-C_{10} Sat. Hydrocarbons C_6-C_{10} Mono & Di-olefins	0.031	ND	ND
Isopropenylbenzene	0.25	ND	ND
Acetophenone	0.17	ND	ND
2-Phenyl-2-propanol	0.70	ND	ND
Carbon Monoxide	0.03	0.002	0.004
Methane	0.03	ND	0.01

ND - Not detected

Table XLVI

GAS-OFF PRODUCTS FROM PRC-1201Q SEALER

AF Serial No. 751

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.26	0.61	0.72
C_5-C_6 Sat. Hydrocarbons	0.06	0.26	0.18
Ethanol	0.80	4.9	1.7
Benzene	0.049	0.025	0.036
n-Propanol	0.022	0.015	0.025
Toluene	211	464	255
Xylene	0.35	0.52	0.45
Unidentified Component (M.W. = 166)	0.05*	ND	ND
Carbon Monoxide	0.07	0.01	0.03
Methane	0.008	ND	0.001

ND - Not detected

*Estimated

Table XLVII

GAS-OFF PRODUCTS FROM F55AP14 EPOXY ENAMEL

AF Serial No. 761

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.24	ND	ND
Methylacetate	0.20	ND	ND
2-Butanone	40.8	11.4	18.6
4-Methyl-2-pentanone	80.0	3.6	5.2
Toluene	40.2	1.6	2.8
n-Butanol	3.9	0.01	0.06
Xylenes	53.2	2.4	2.6
C_3 Alkylbenzenes	16.0	0.19	0.31
2-n-Propylthiophene	13.0	0.18	0.28
C_4 Alkylbenzenes	0.35	ND	ND
Carbon Monoxide	0.14	0.004	0.006
Methane	0.03	ND	ND

ND - Not detected

Table XLVIII

GAS-OFF PRODUCTS FROM SYLGARD 182 POTTING COMPOUND

AF Serial No. 786

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C_4-C_6 Sat. Hydrocarbons	0.069	0.027	0.036
Acetone	0.062	0.010	ND
Silicone Oil (3 types)	0.65	0.10	0.046
Benzene	0.013	ND	0.006
Toluene	0.21	0.094	0.046
Trimethylsilanol	0.055	ND	ND
Xylenes	1.1	0.53	0.36
Carbon Monoxide	0.02	ND	0.006
Methane	0.11	ND	0.02

ND - Not detected

Table XLIX

GAS-OFF PRODUCTS FROM DC93-500 PART A & B

AF Serial No. 955

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)
Acetone	0.012	ND
Benzene	0.87	0.41
Silicone Oil	0.051	0.045
Toluene	0.006	ND
Carbon Monoxide	0.008	ND
Methane	0.50	0.03

ND - Not detected

Table L

GAS-OFF PRODUCTS FROM DAC-026

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.009	0.005	ND
Silicone Oil (4 types)	2.0	1.5	1.6
Trimethylsilanol	0.29	0.012	0.005
2,4-Pentanedione	0.07*	ND	ND
Carbon Monoxide	ND	0.002	ND
Methane	0.02	0.03	0.005

ND - Not detected

*Tentative value, subject to interference from low molecular weight siloxane.

Table LI

GAS-OFF PRODUCTS FROM DAC-029

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.073	0.009	ND
Silicone Oil (3 types)	0.93	0.38	0.48
Benzene	0.032	ND	ND
Trimethylsilanol	0.80	0.043	0.043
2,4-Pentanedione	0.11*	ND	ND
Carbon Monoxide	ND	ND	0.01
Methane	0.01	0.009	0.03

ND - Not detected

*Tentative value, subject to interference from a low molecular weight siloxane.

Table LII

GAS-OFF PRODUCTS FROM DAC-030

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.011	ND	ND
Silicone Oil (3 types)	0.086	ND	ND
Trimethylsilanol	0.021	ND	ND
Xylenes	0.016	ND	ND
Carbon Monoxide	ND	ND	ND
Methane	ND	ND	ND

ND - Not detected

Table LIII

GAS-OFF PRODUCTS FROM DAC-032

Component	Weight of Component (mg/10 gms Candidate Material) 78 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Toluene	0.011	ND	ND
Xylenes	0.031	ND	ND
Diethoxyethanol	0.091	ND	ND
Carbon Monoxide	0.02	0.001	0.004
Methane	0.06	0.003	0.009

ND - Not detected

Table LIV

GAS-OFF PRODUCTS FROM DAC-033

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.031	ND	ND
Butyraldehyde	0.031	ND	ND
Ethanol	0.030	ND	ND
n-Propanol	0.004	ND	ND
2-Butanol	0.004	ND	ND
Toluene	0.005	ND	ND
n-Butanol	0.28	0.003	ND
2-n-Butoxyethanol	0.11	ND	ND
Carbon Monoxide	0.01	ND	ND
Methane	0.01	ND	ND

ND - Not detected

Table LV

GAS-OFF PRODUCTS FROM DAC-037

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.053	ND	ND
C_9 Saturated Hydrocarbon	0.016	ND	ND
Benzene	0.010	ND	0.012
Toluene	0.95	ND	0.019
Xylene	0.82	ND	0.029
Carbon Monoxide	0.002	ND	0.007
Methane	0.02	ND	0.03

ND - Not detected

Table LVI

GAS-OFF PRODUCTS FROM DAC-039

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.016	ND	ND
Silicone Oil (2 types)	0.079	ND	0.005
C_9 Saturated Hydrocarbon	0.16	0.08	0.18
Benzene	0.061	0.007	0.006
2-Butanol	0.018	0.004	0.006
Trimethylsilanol	0.067	ND	ND
Xylenes	0.018	ND	ND
Carbon Monoxide	0.005	0.006	0.01
Methane	0.03	0.01	0.03

ND - Not detected

Table LVII

GAS-OFF PRODUCTS FROM DAC-042

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.039	ND	ND
n-Butanol	0.028	ND	ND
2-n-Butoxyethanol	0.020	ND	ND
Carbon Monoxide	0.020	0.005	0.01
Methane	0.01	0.03	0.03

ND - Not detected

Table LVIII

GAS-OFF PRODUCTS FROM DAC-044

Component	Weight of Component (mg/10 gms Candidate Material) 72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.019	ND	ND
Silicone Oil (2 types)	0.026	ND	ND
C_9 Saturated Hydrocarbon	0.009	ND	ND
Trimethylsilanol	0.090	0.004	ND
Toluene	0.028	0.003	ND
Xylenes	0.21	ND	ND
C_3-C_4 Alkylbenzenes	0.061	ND	ND
Acetic Acid	0.072	ND	ND
Carbon Monoxide	0.008	ND	0.002
Methane	0.009	ND	0.02

ND - Not detected

APPENDIX III

REPRESENTATIVE GAS CHROMATOGRAMS

FOR

GAS-OFF EXPERIMENTS

The gas chromatograms shown in this appendix were obtained on an F&M Scientific Corporation Model 810 Research Gas Chromatograph. Instrument conditions and column specifications are listed in Table LIX. Since retention times tended to shift somewhat due to column aging, a standard mixture was used as a day-to-day reference. The first peak appearing in each chromatogram is air.

The gas chromatograms are representative of a particular candidate material. Comparison of peak intensities in chromatograms for different candidate materials should be made with care, since sensitivity factors and quantities of atmosphere taken for analysis vary.

Chromatograms appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

Table LIX

GAS CHROMATOGRAPHIC INSTRUMENT CONDITIONS

All samples were analyzed using a flame ionization detector and a F&M Model 810 Research Gas Chromatograph in a single column and single detector mode.

Instrument Conditions

Column: 20-ft x 1/4-in. O.D. Stainless Steel, 20% Triton X-305 on 60/80 mesh Gas Chrom Z.

Column Temperature: programmed 50°-170°C @ 8°C/min.

Detector Temperature: 300°C

Injection Port Temperature: 250°C

Flow Split: none

Flow Rate: 60 ml/min. He

Range: 10

Attenuation: X8, or as noted

Sample Size: 50 cc of gas

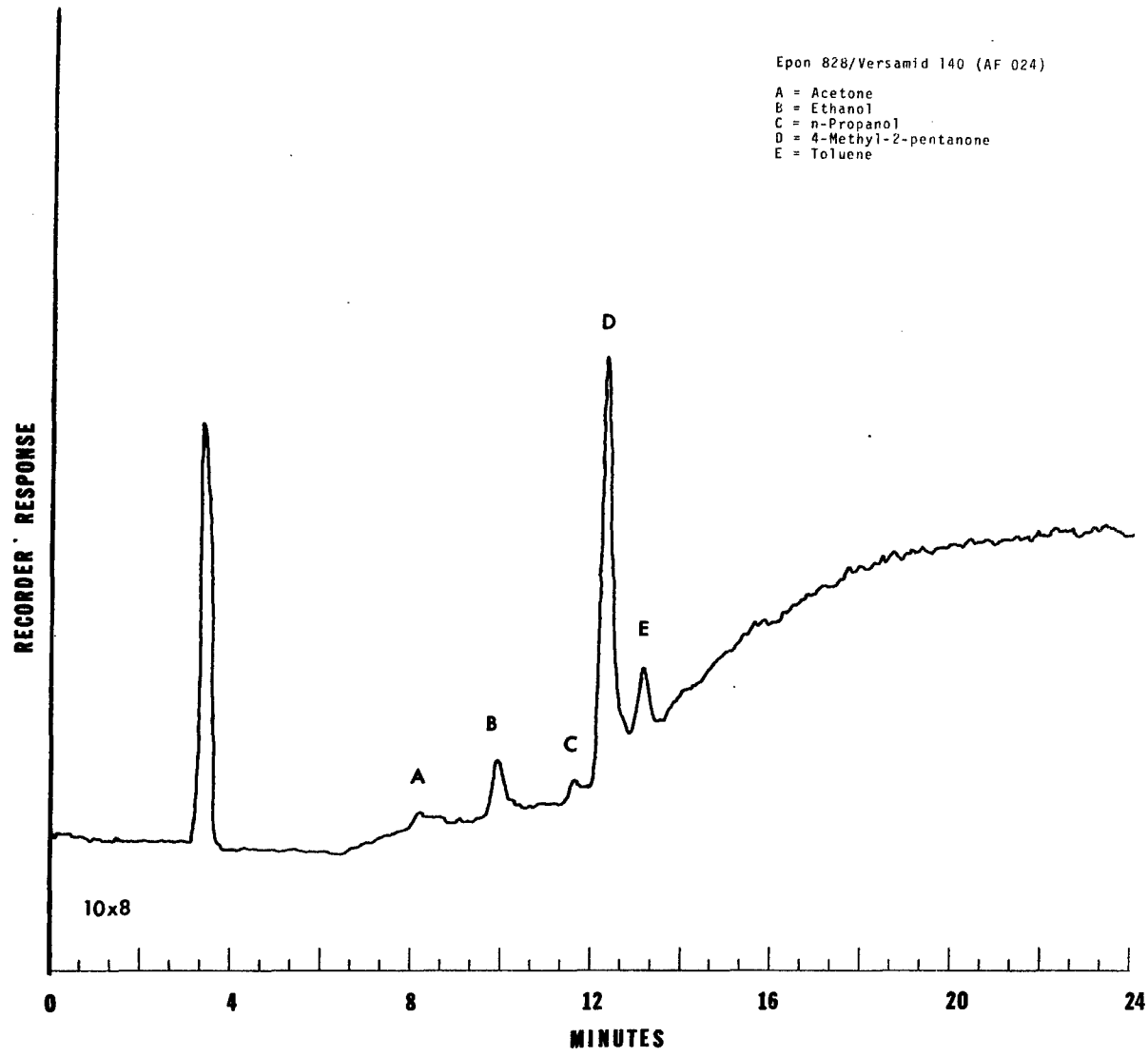

Figure 63. Gas Chromatogram of Gas-Off Products From Epon 828/Versamid 140 (AF 024) (72 Hours @ 68°C).

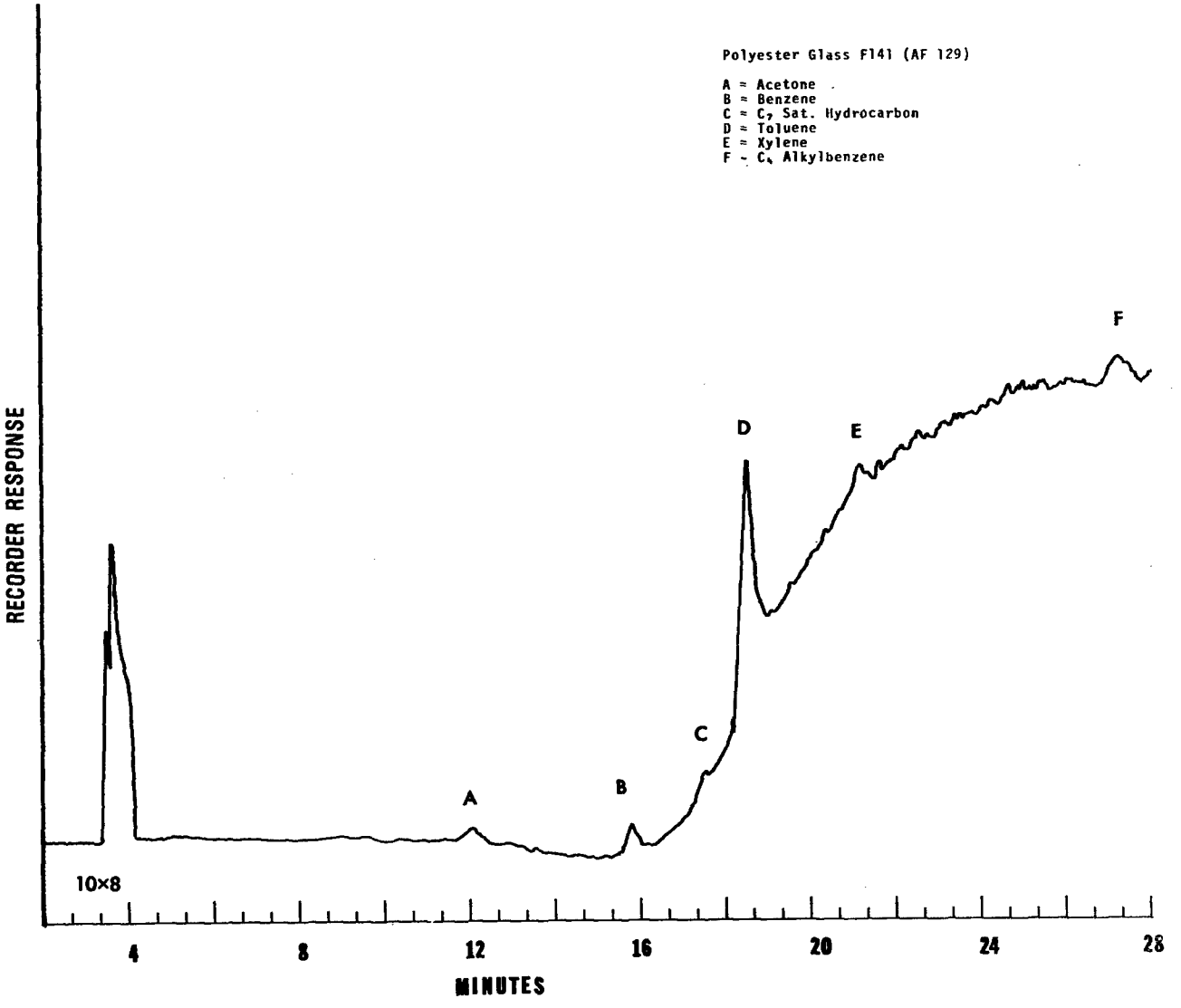

Figure 64. Gas Chromatogram of Gas-Off Products From Polyester Glass F141 (AF 129) (72 Hours @ 68°C).

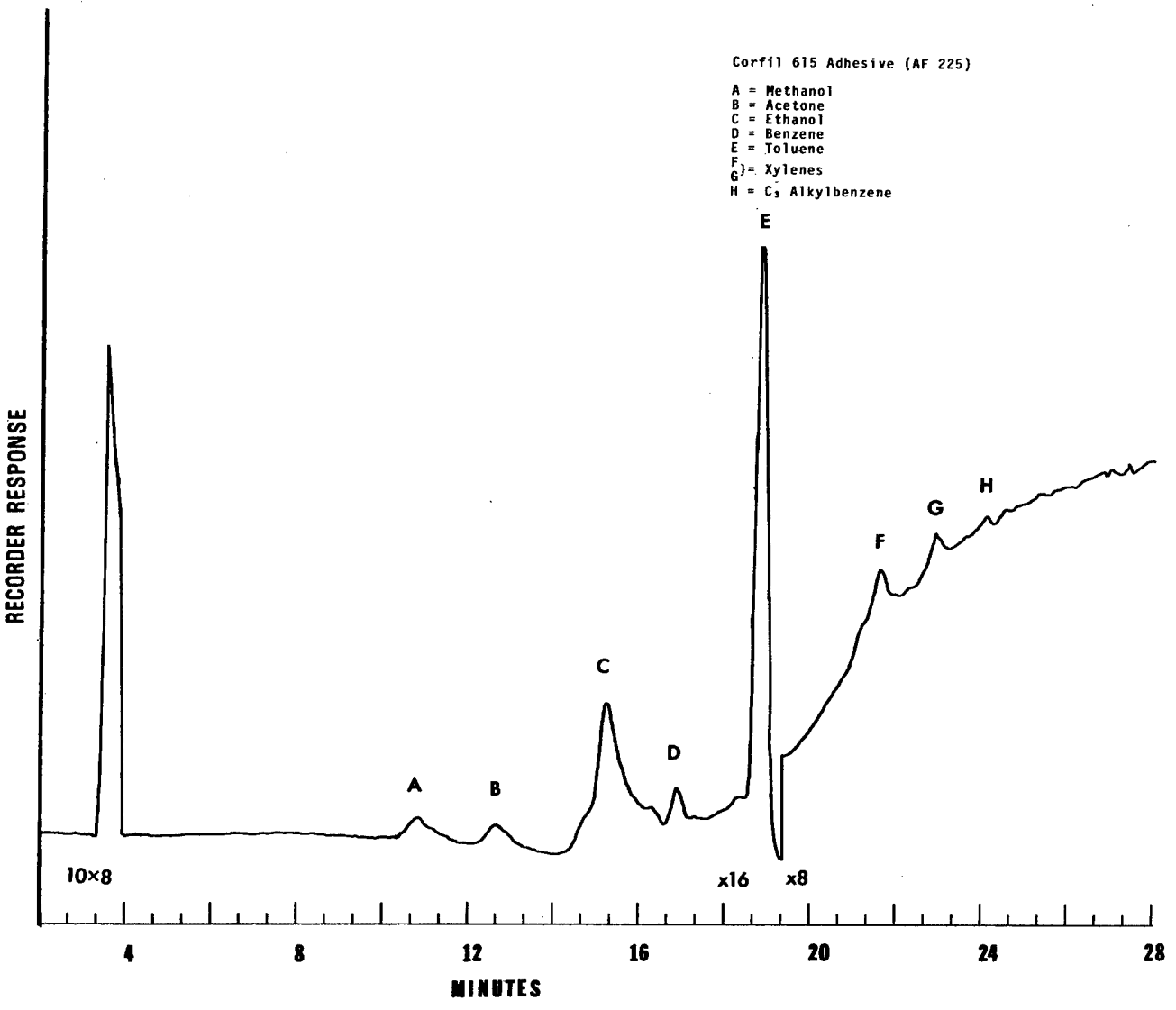

Figure 65. Gas Chromatogram of Gas-Off Products From Corfil 615 Adhesive (AF 225) (72 Hours @ 68°C).

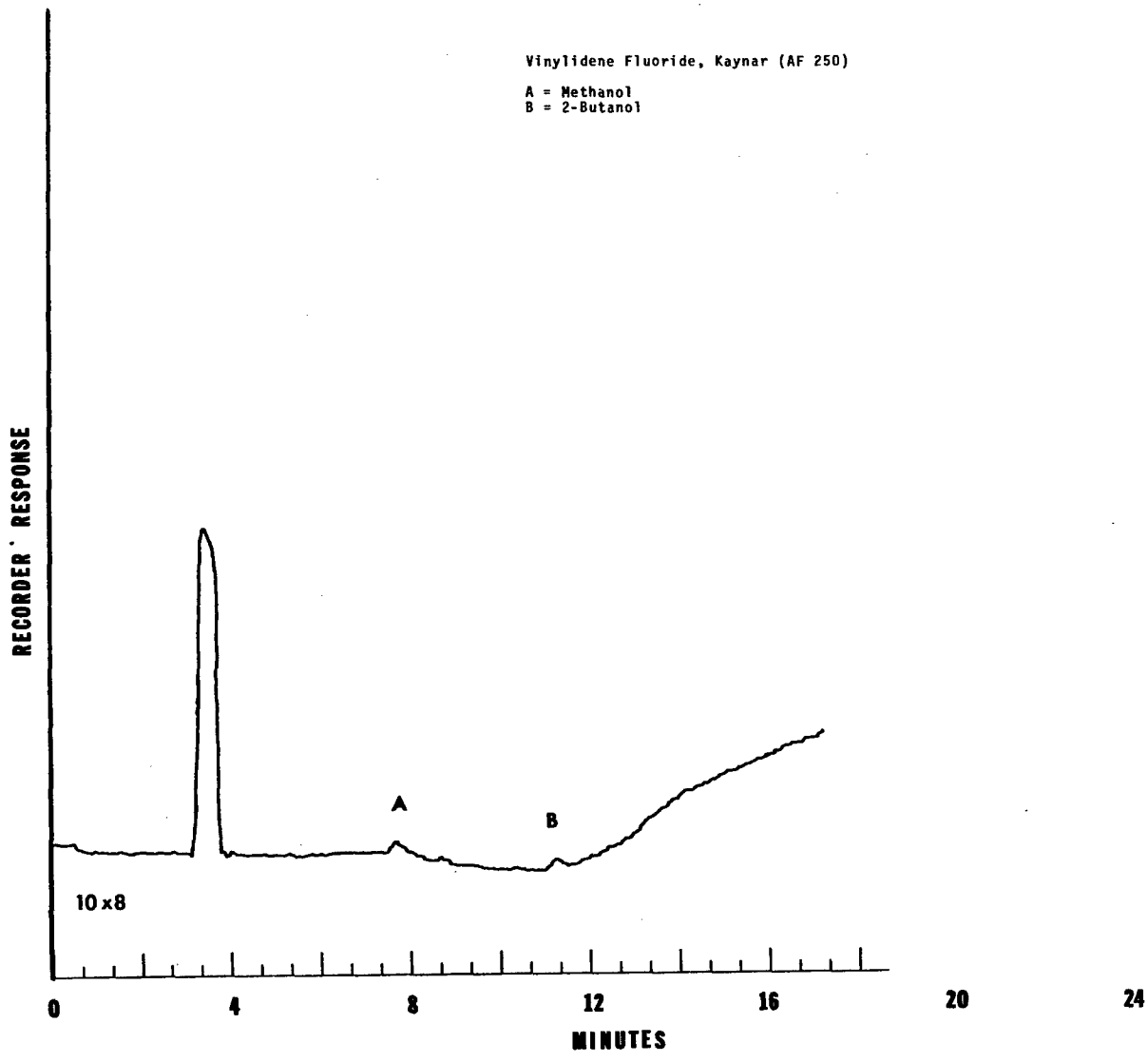

Figure 66. Gas Chromatogram of Gas-Off Products From Vinylidene Fluoride, Kaynar (AF 250) (72 Hours @ 68°C).

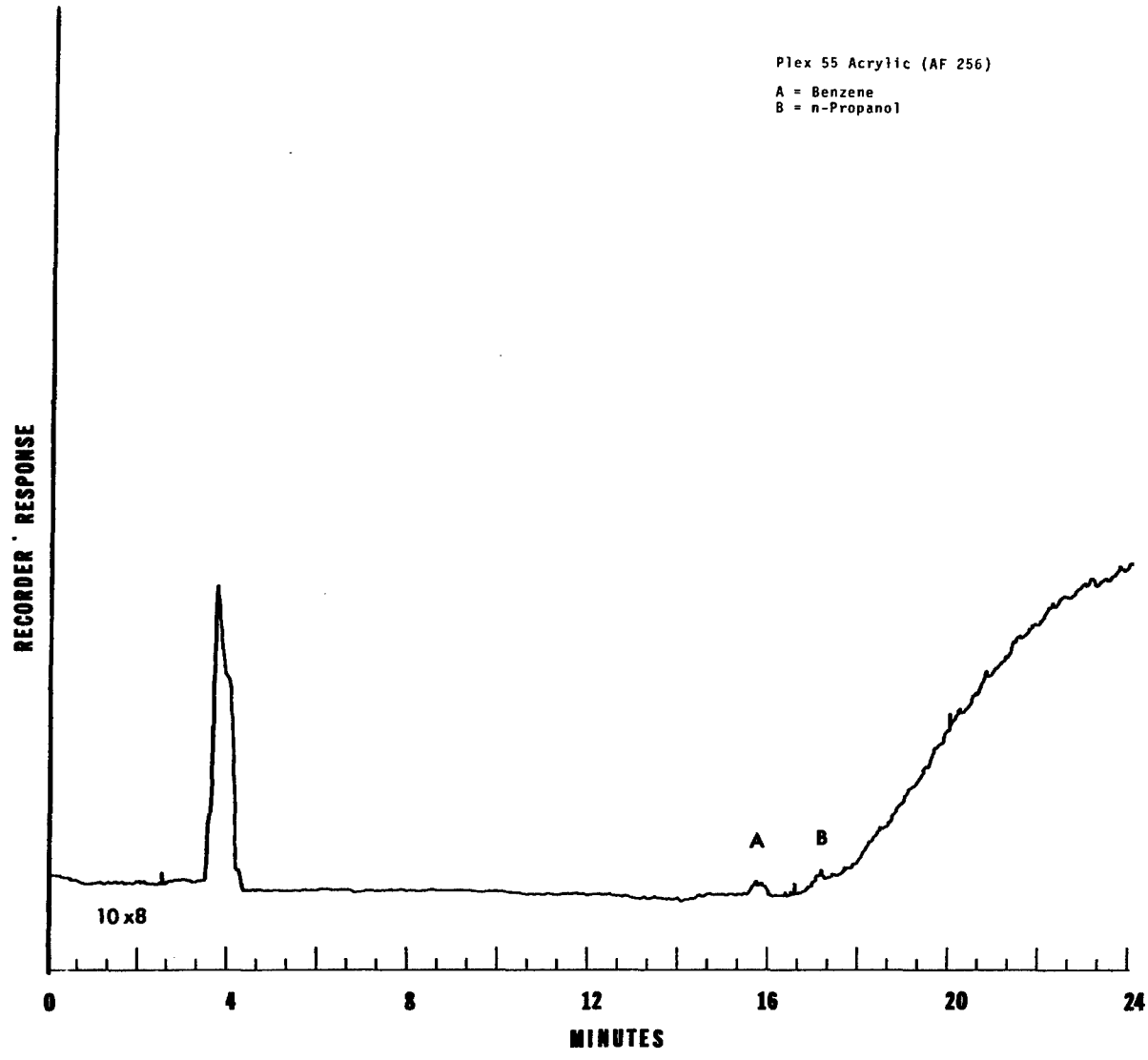

Figure 67. Gas Chromatogram of Gas-Off Products From Plex 55 Acrylic (AF 256) (72 Hours @ 68°C).

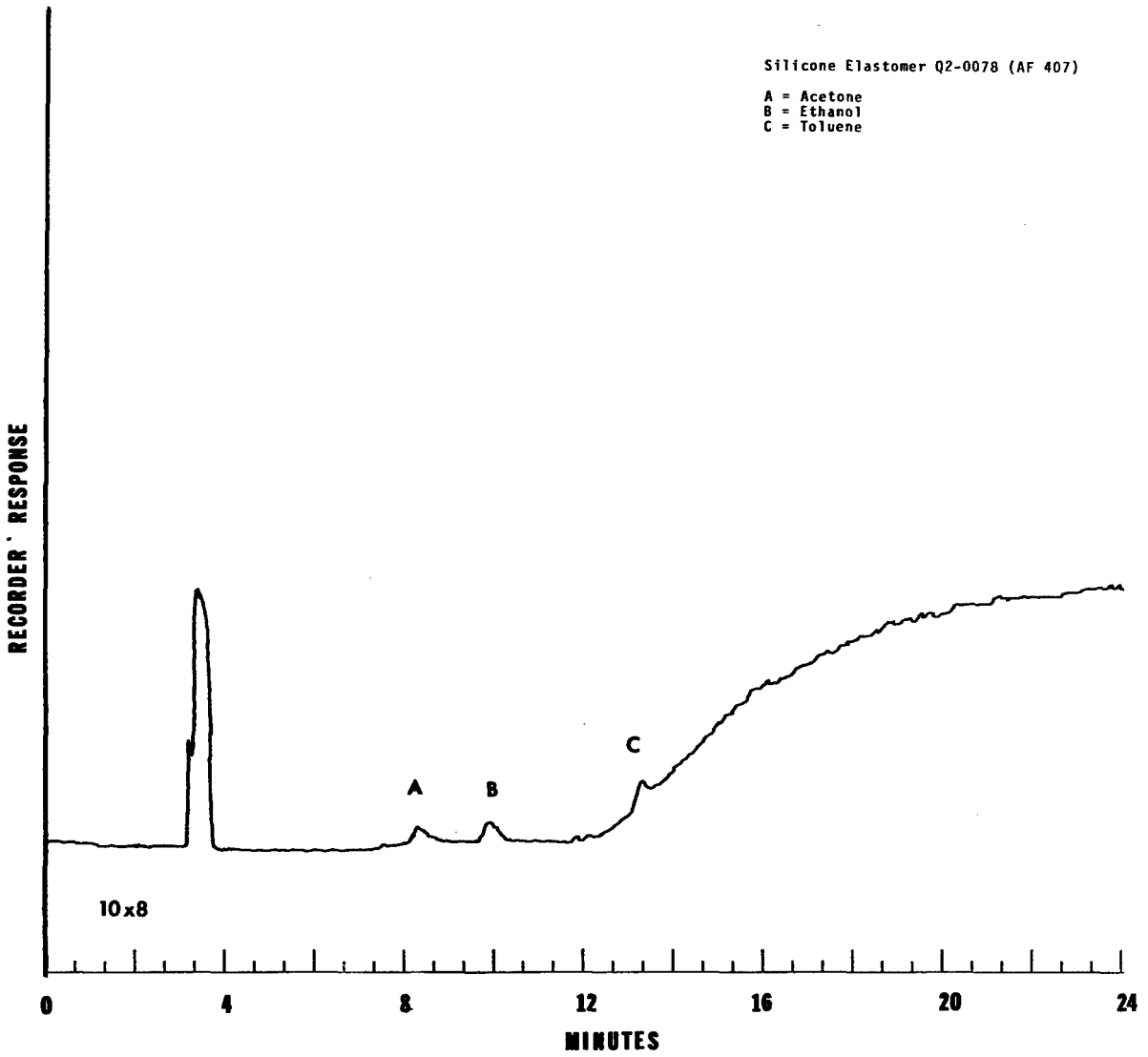

Figure 68. Gas Chromatogram of Gas-Off Products From Silicone Elastomer Q2-0078 (AF 407) (72 Hours @ 68°C).

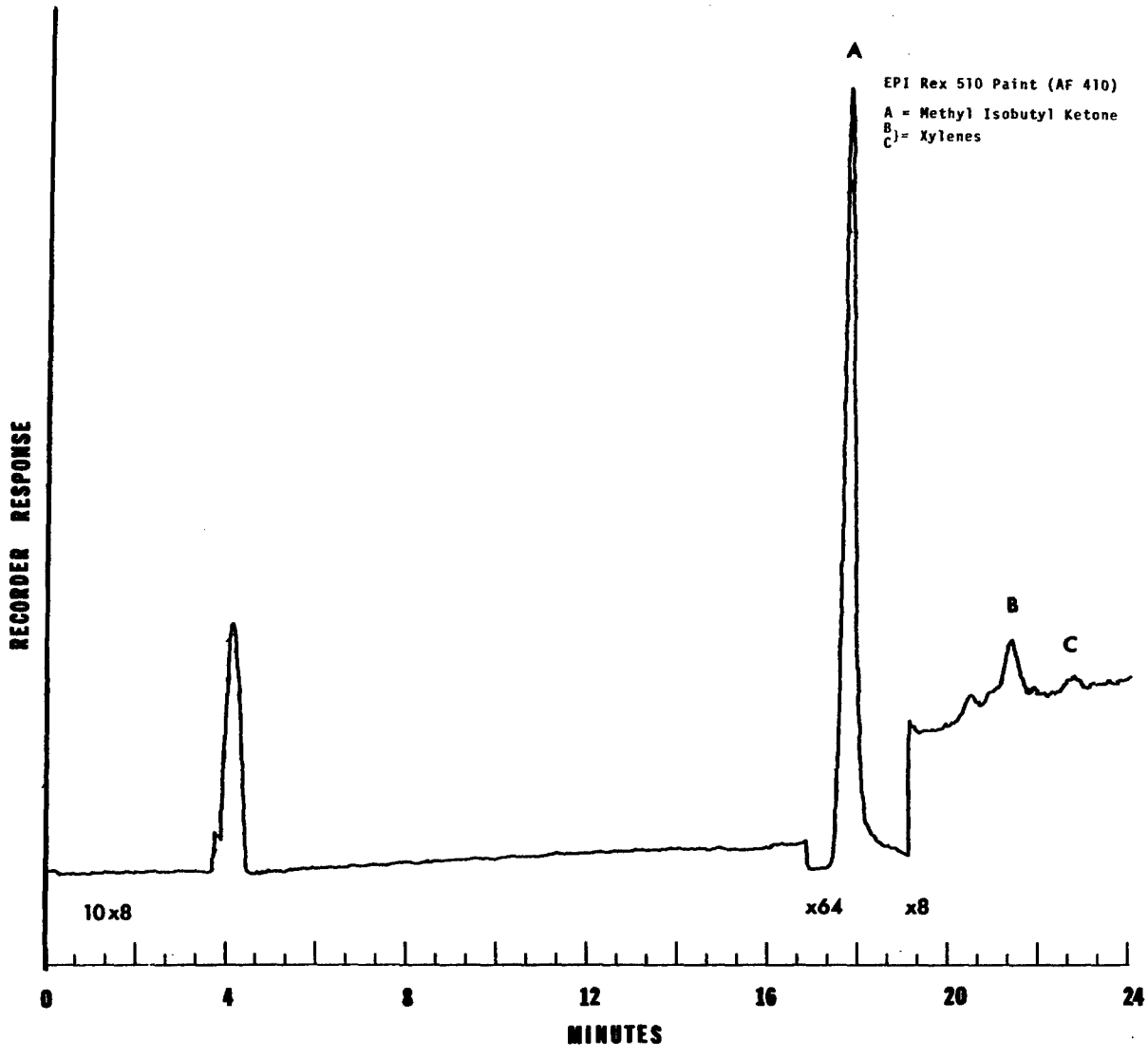

Figure 69. Gas Chromatogram of Gas-Off Products From EPI Rex 510 Paint (AF 410) (72 Hours @ 68°C).

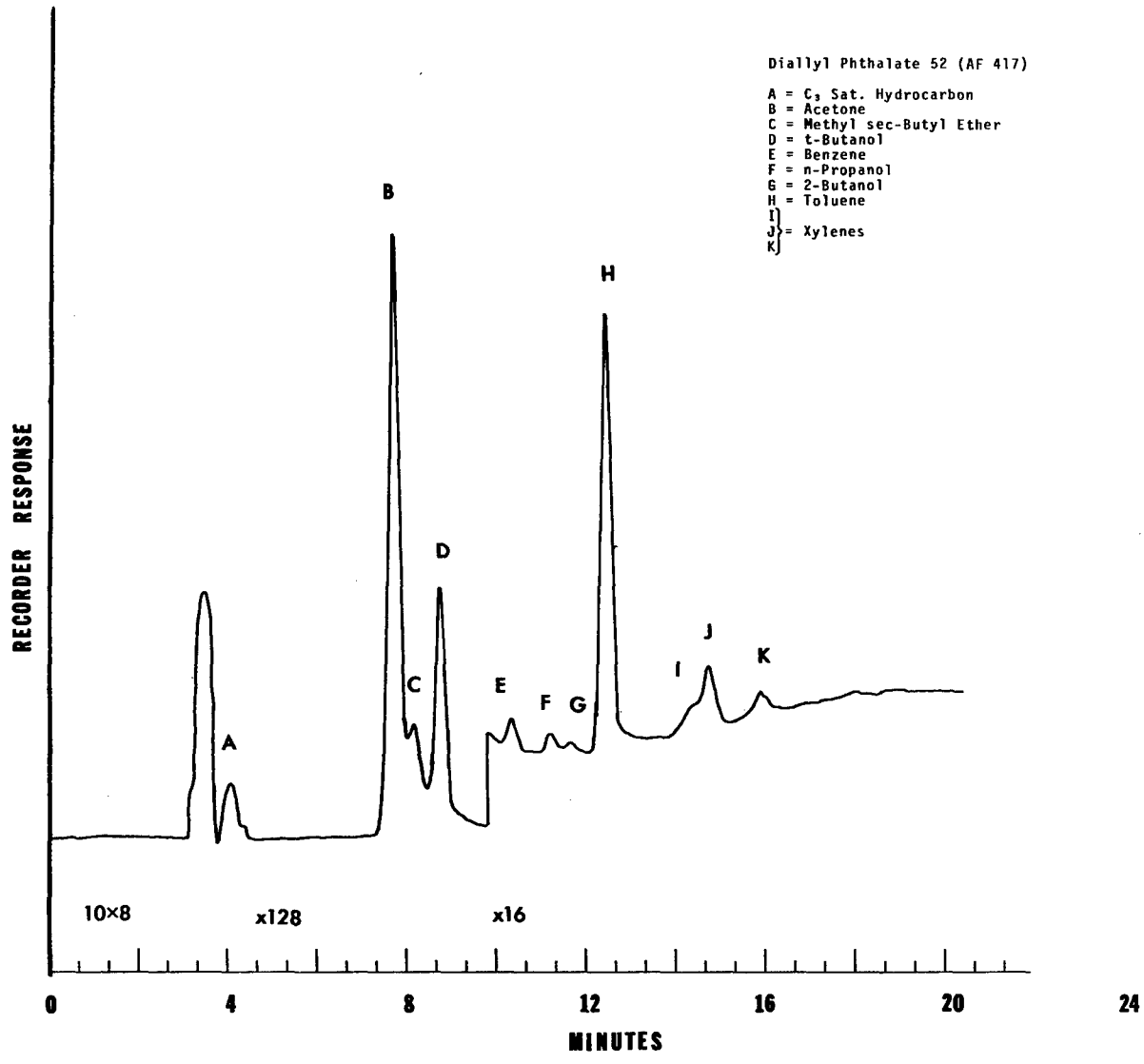

Figure 70. Gas Chromatogram of Gas-Off Products From Diallyl Phthalate 52 (AF 417) (72 Hours @ 68°C).

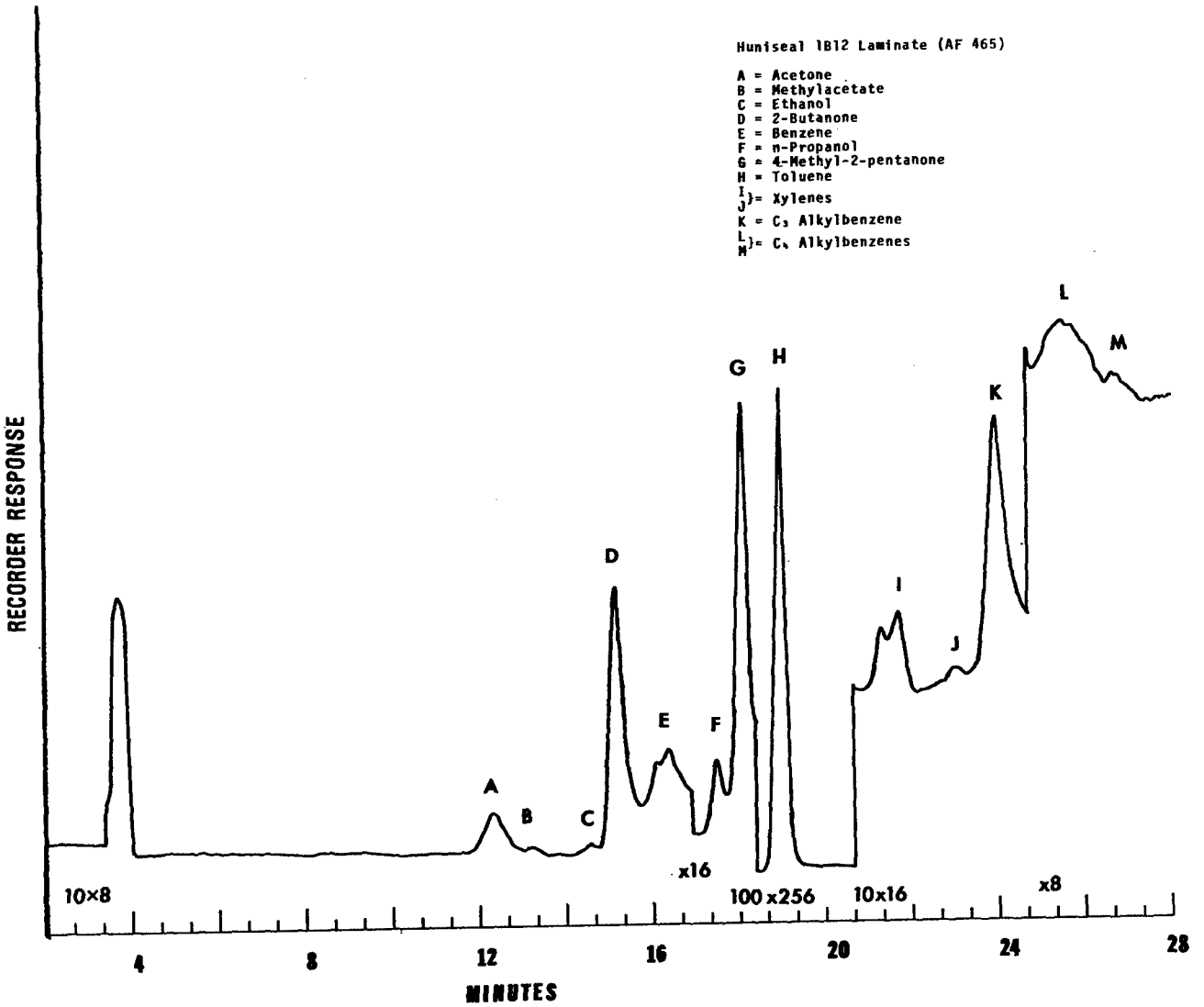

Figure 71. Gas Chromatogram of Gas-Off Products From Huniseal 1B12 Laminate (AF 465) (72 Hours @ 68°C).

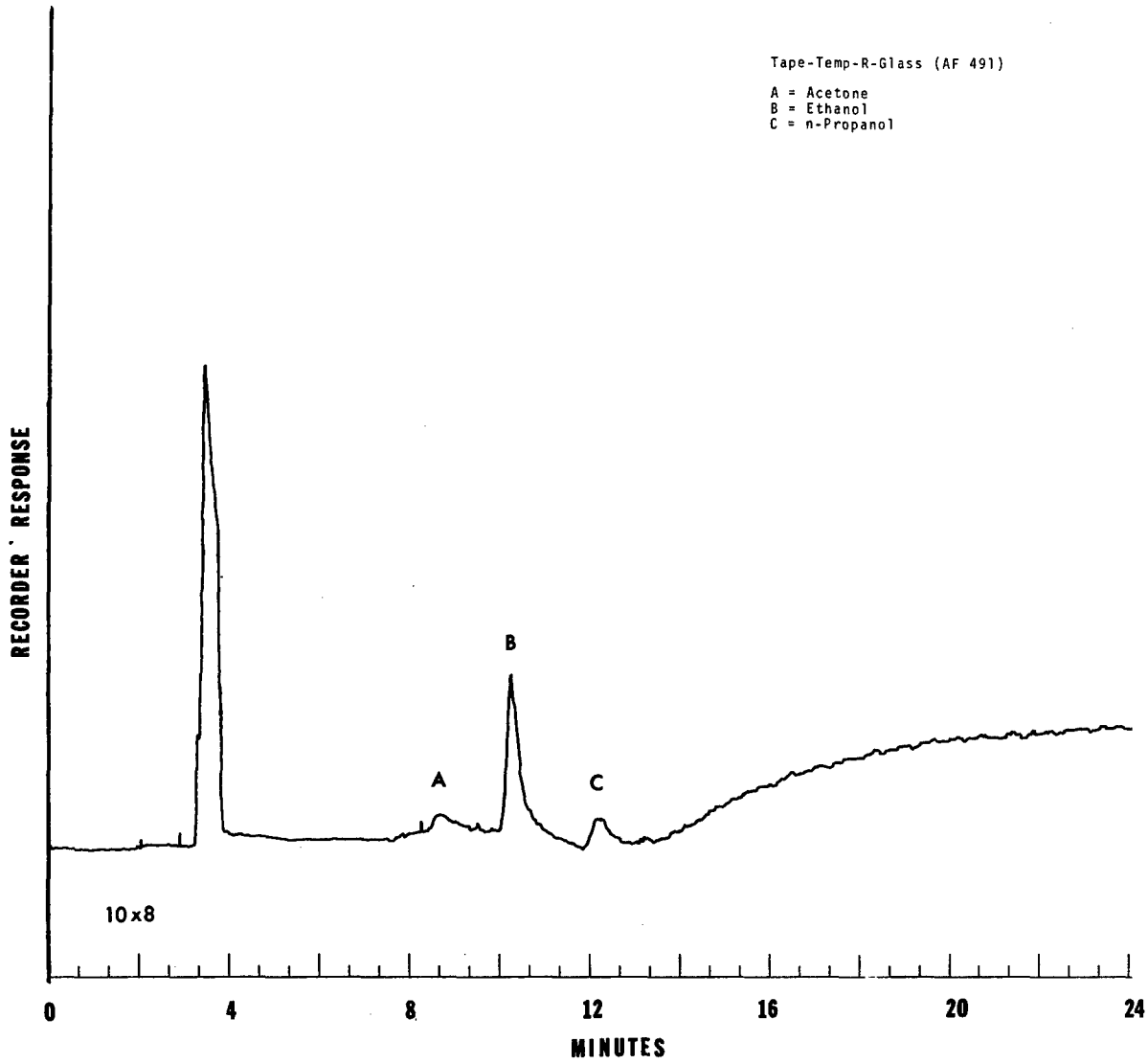

Figure 72. Gas Chromatogram of Gas-Off Products From Tape-Temp-R-Glass (AF 491) (72 Hours @ 68°C).

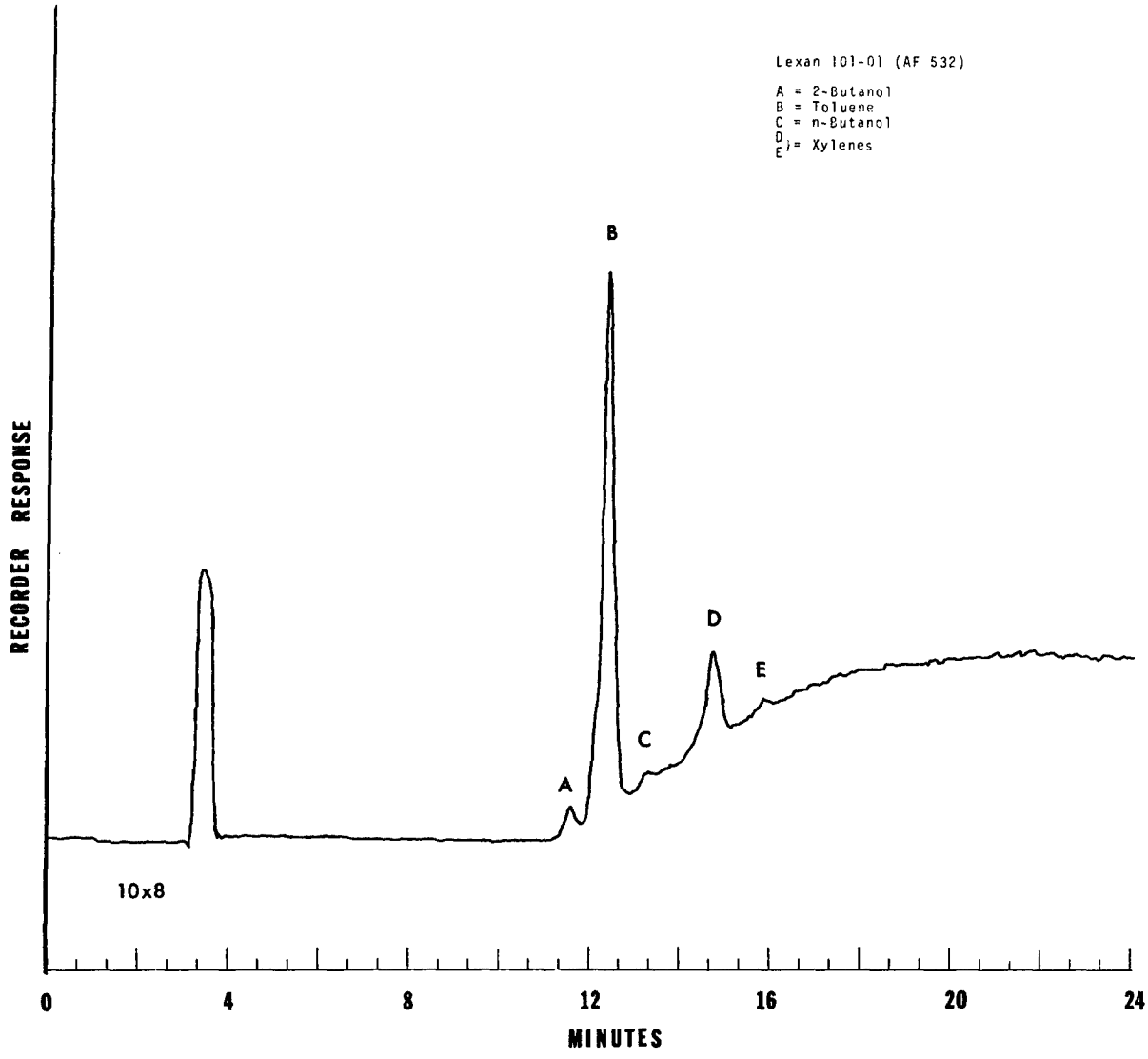

Figure 73. Gas Chromatogram of Gas-Off Products From Lexan 101-01 (AF 532) (72 Hours @ 68°C).

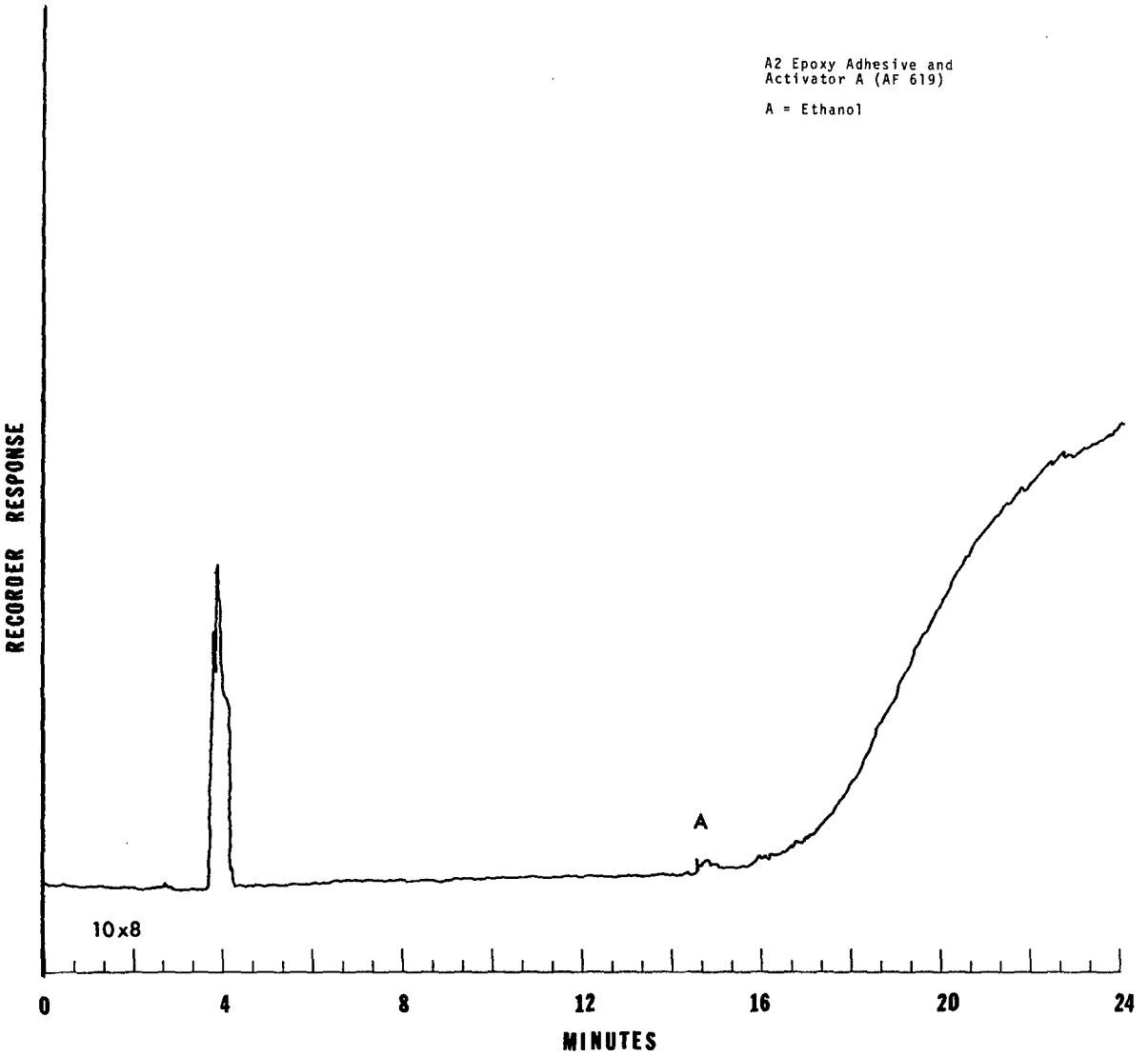

Figure 74. Gas Chromatogram of Gas-Off Products From A2 Epoxy Adhesive and Activator A (AF 619) (72 Hours @ 68°C).

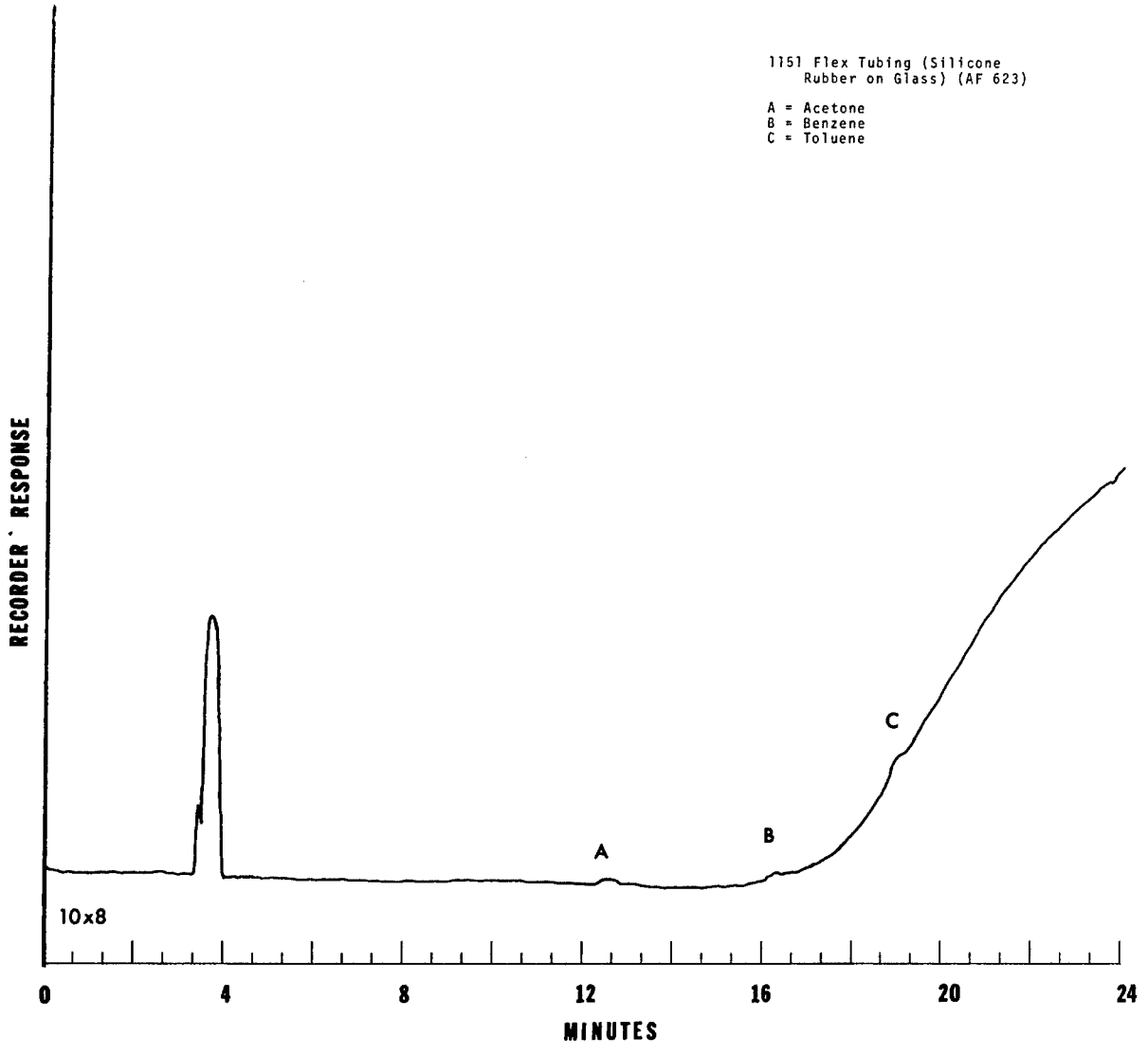

Figure 75. Gas Chromatogram of Gas-Off Products From 1151 Flex Tubing (Silicone Rubber on Glass) (AF 623) (72 Hours @ 68°C).

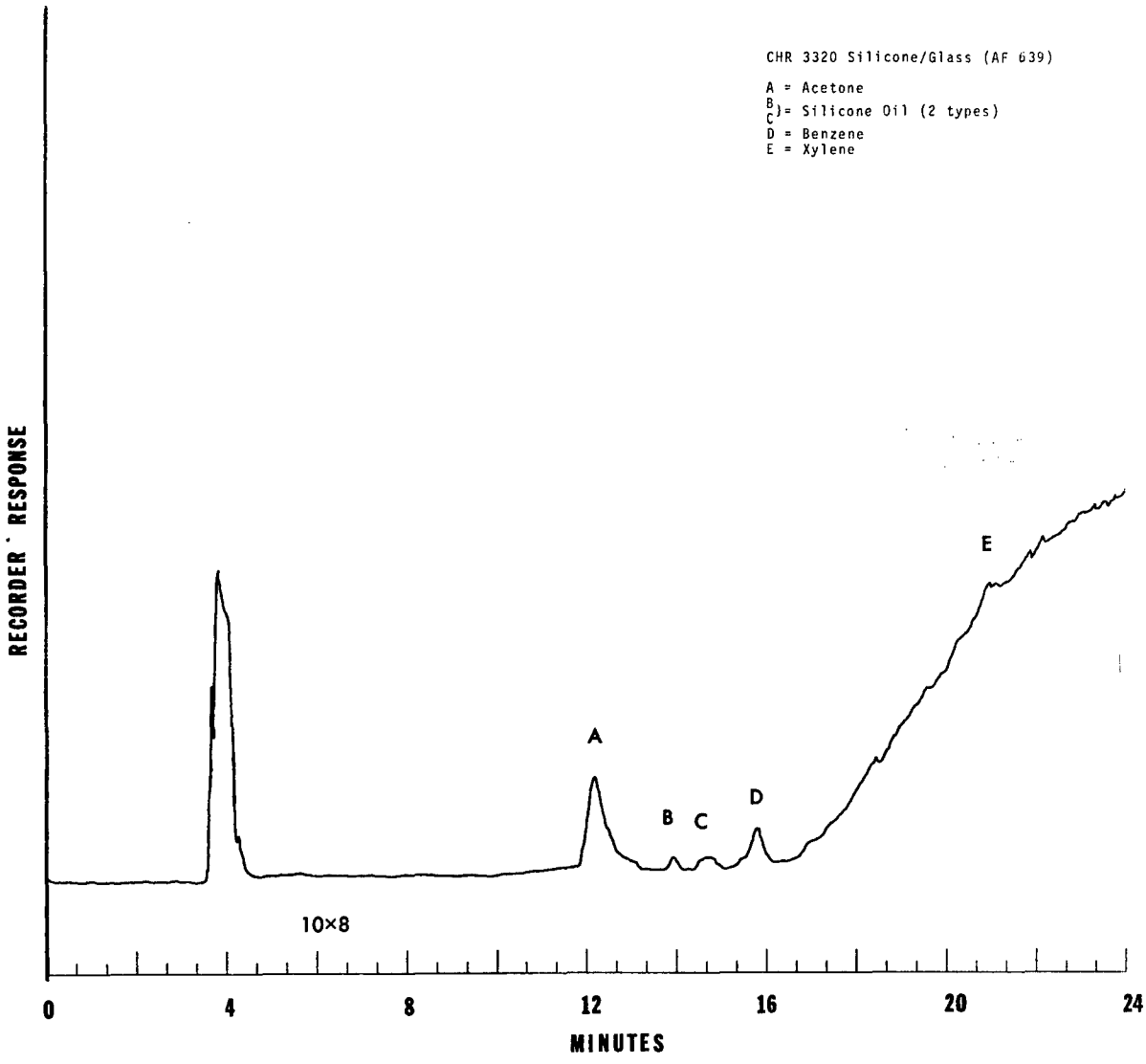

Figure 76. Gas Chromatogram of Gas-Off Products From CHR 3320 Silicone/Glass (AF 639) (72 Hours @ 68°C).

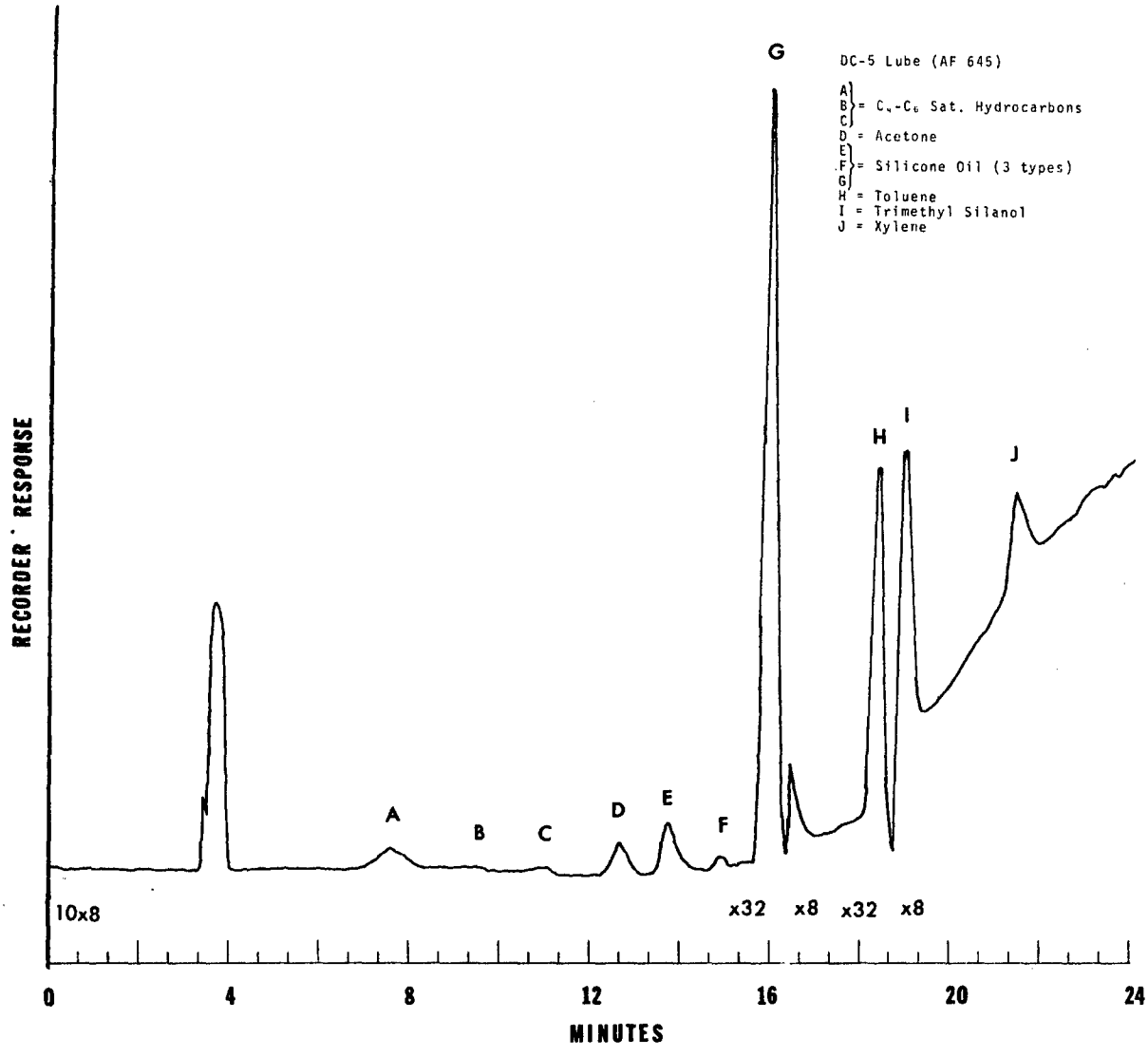

Figure 77. Gas Chromatogram of Gas-Off Products From DC-5 Lube (AF 645) (72 Hours @ 68°C).

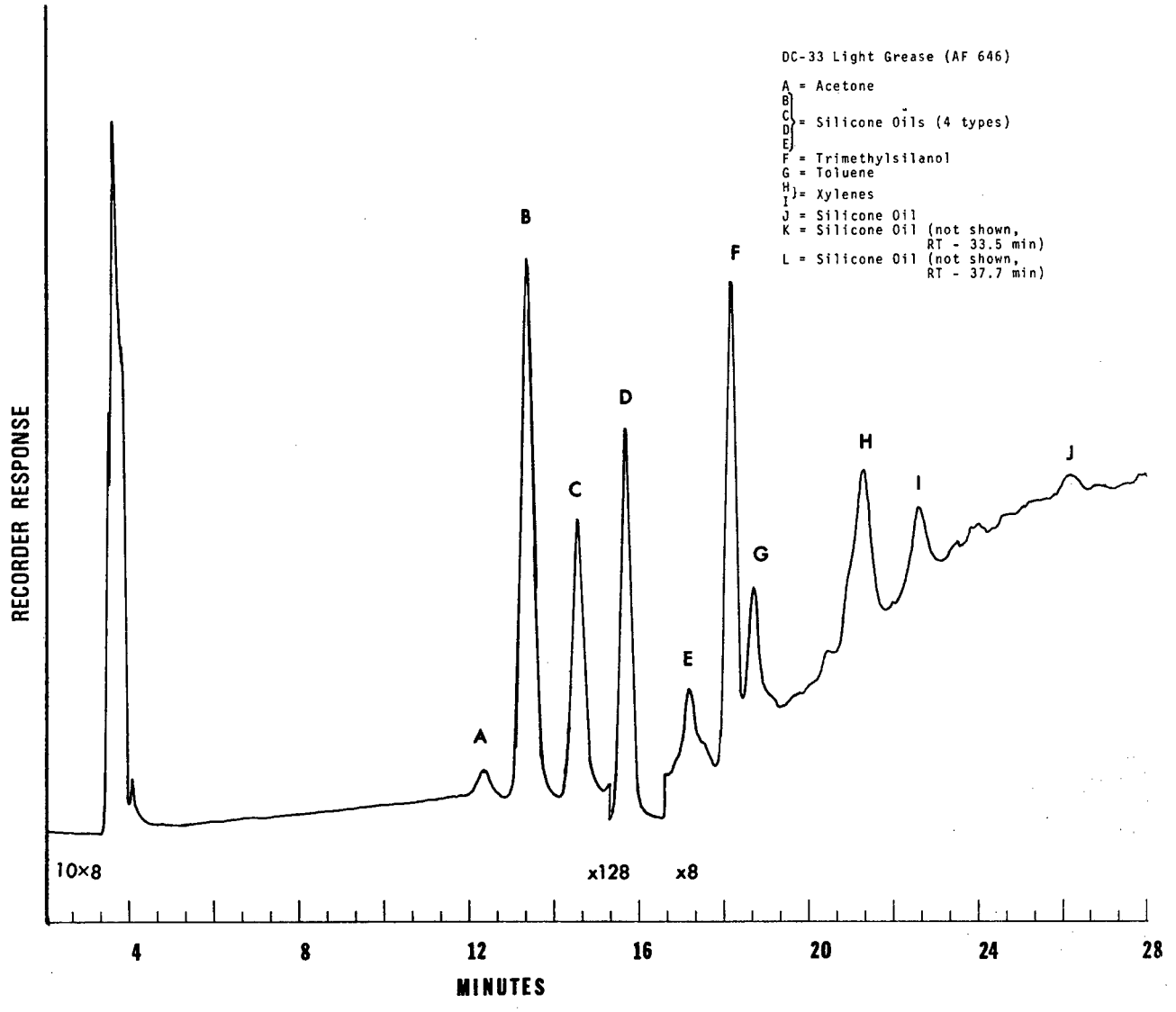

Figure 78. Gas Chromatogram of Gas-Off Products From DC-33 Light Grease (AF 646) (72 Hours @ 68°C).

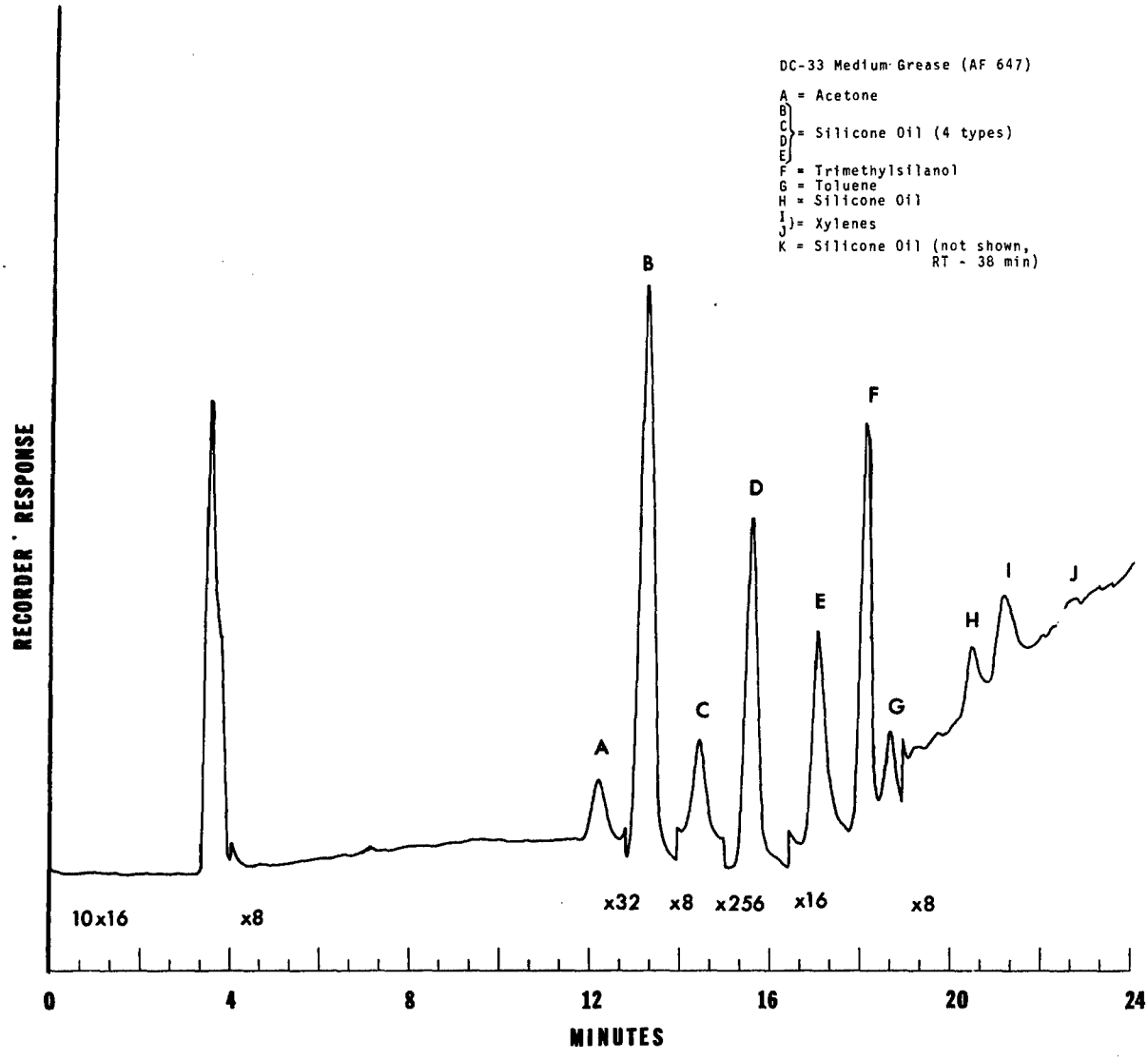

Figure 79. Gas Chromatogram of Gas-Off Products From DC-33 Medium Grease (AF 647) (72 Hours @ 68°C).

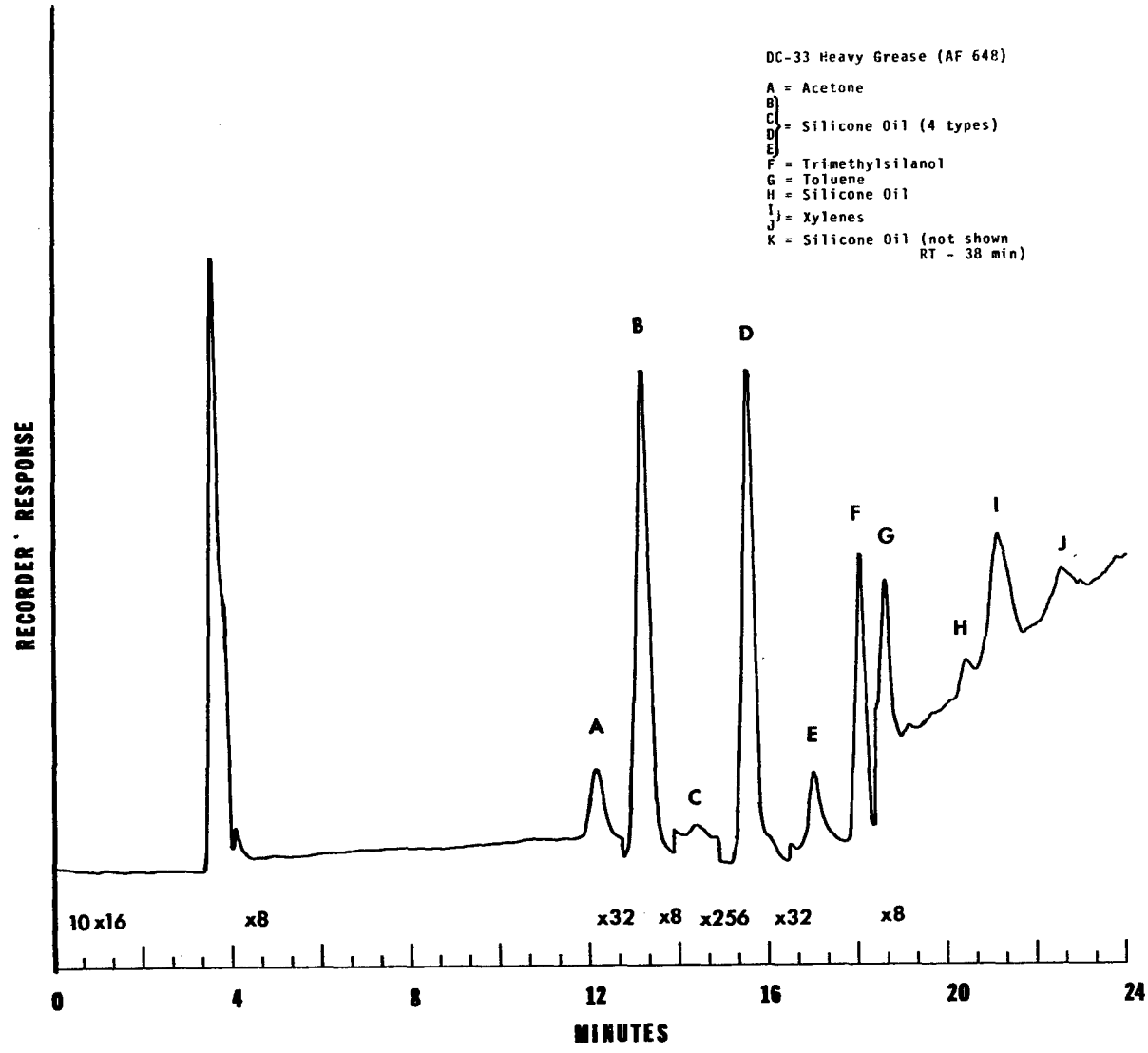

Figure 80. Gas Chromatogram of Gas-Off Products From DC-33 Heavy Grease (AF 648) (72 Hours @ 68°C).

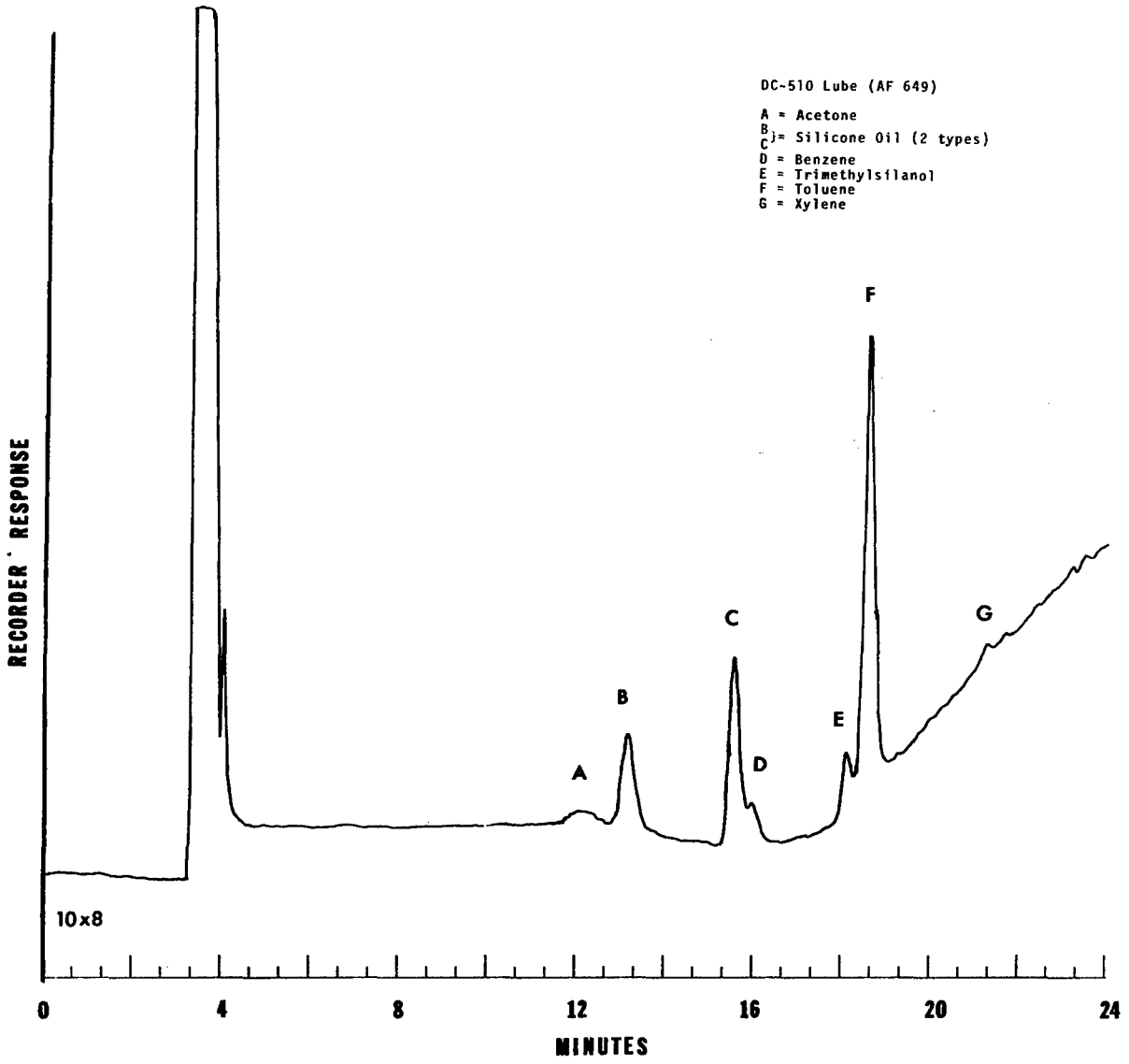

Figure 81. Gas Chromatogram of Gas-Off Products From DC-510 Lube (AF 649) (72 Hours @ 68°C).

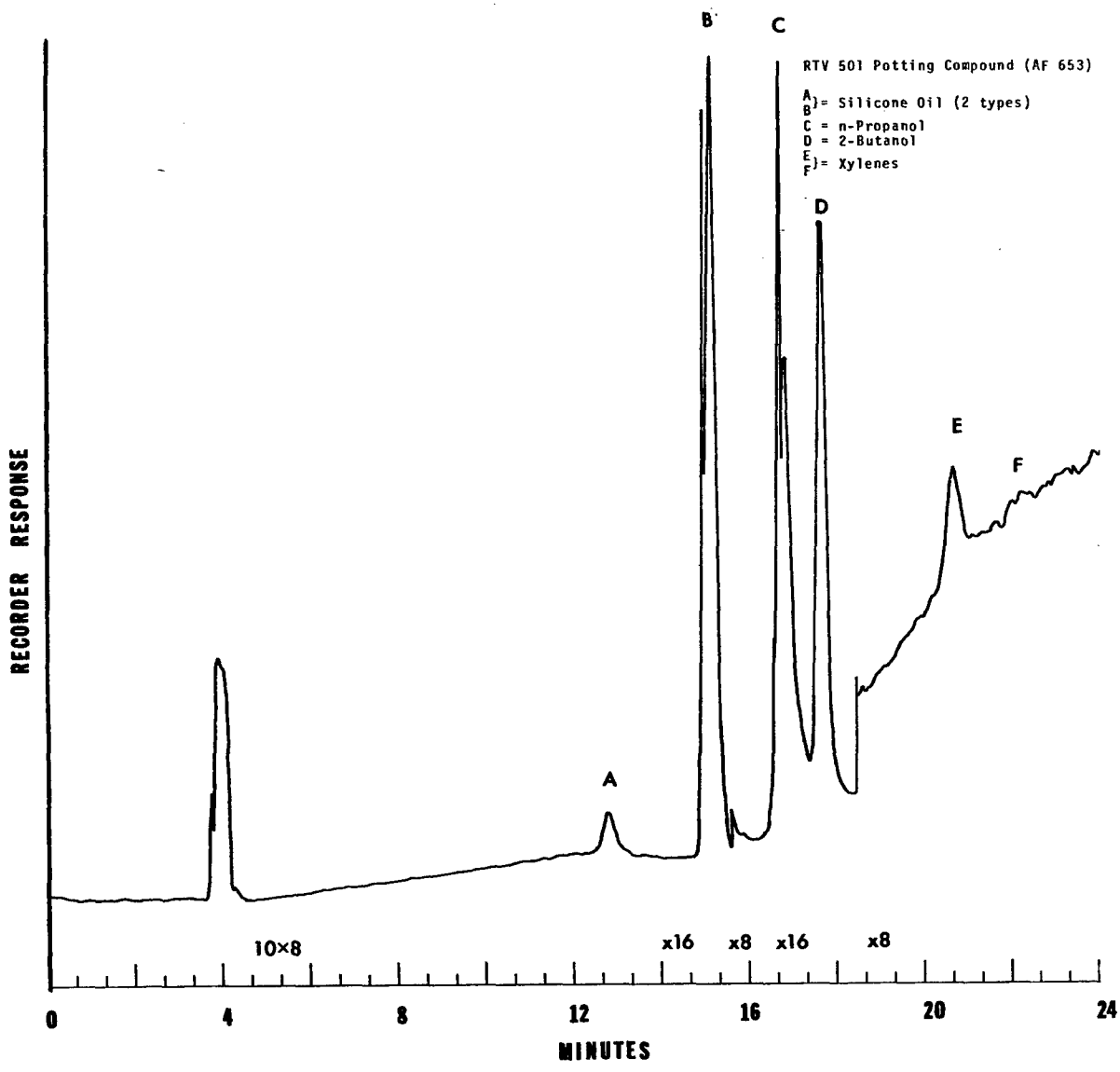

Figure 82. Gas Chromatogram of Gas-Off Products From RTV 501 Potting Compound (AF 653) (72 Hours @ 68°C).

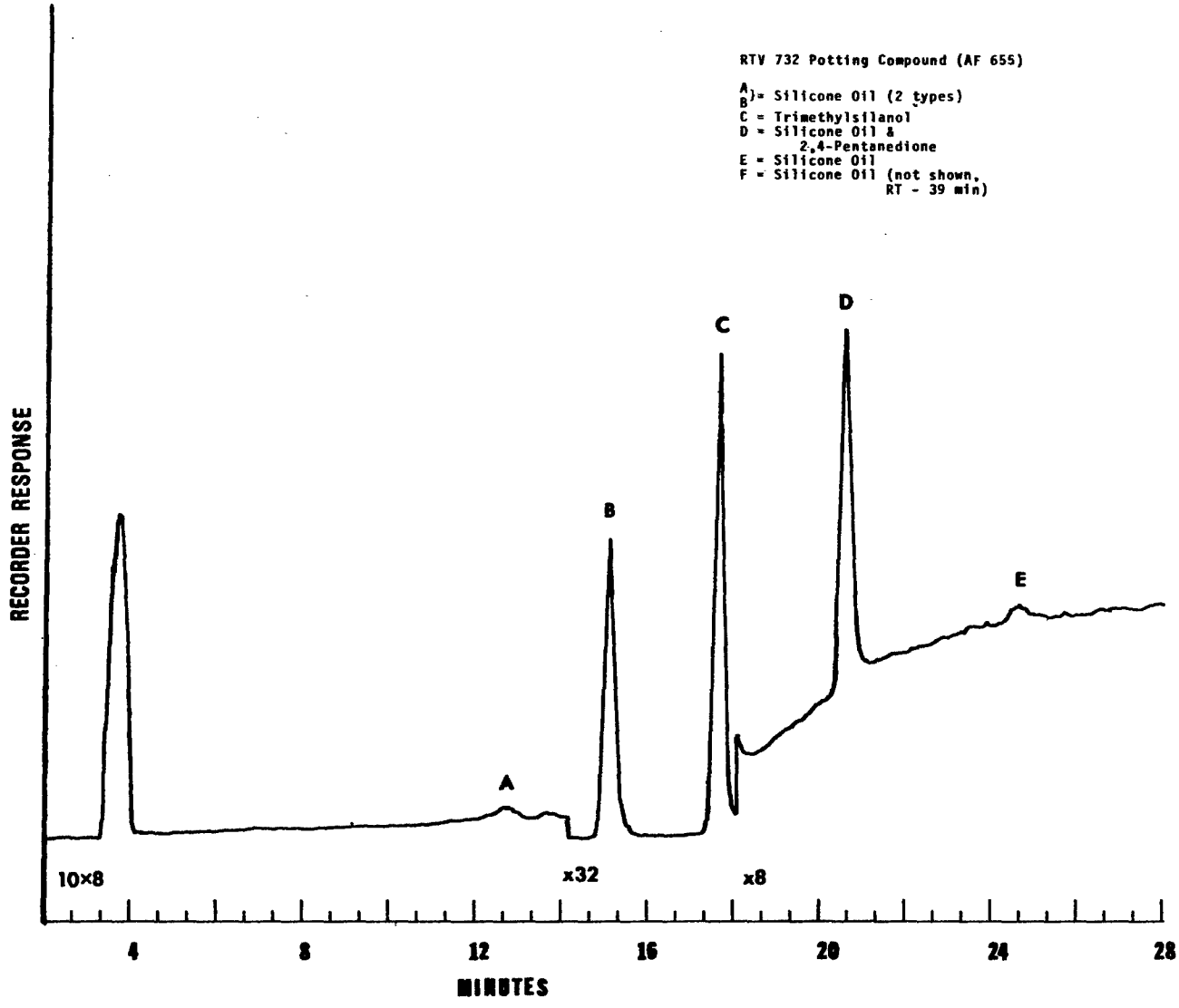

Figure 83. Gas Chromatogram of Gas-Off Products From RTV 732 Potting Compound (AF 655) (72 Hours @ 68°C).

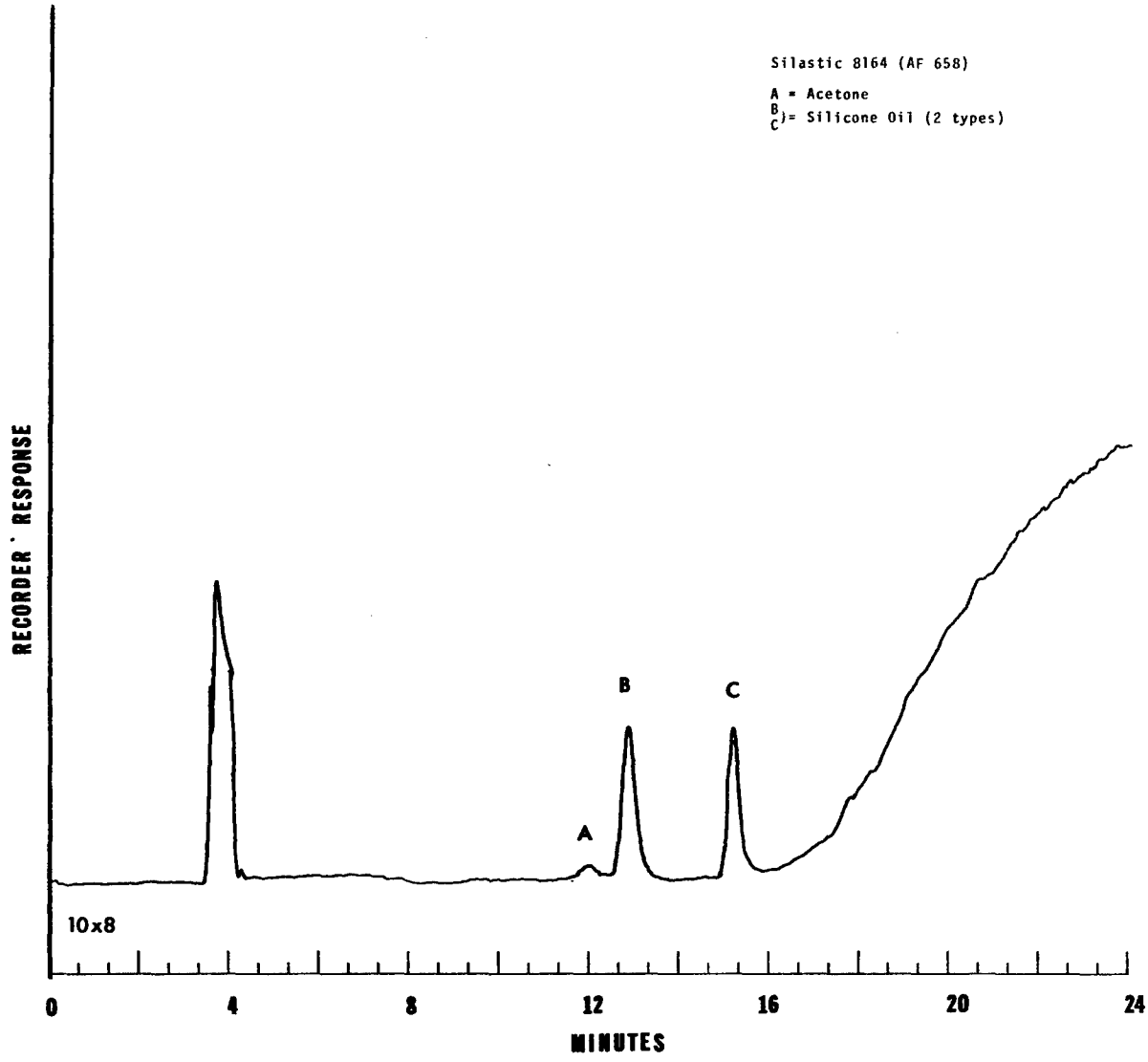

Figure 84. Gas Chromatogram of Gas-Off Products From Silastic 8164 (AF 658) (72 Hours @ 68°C).

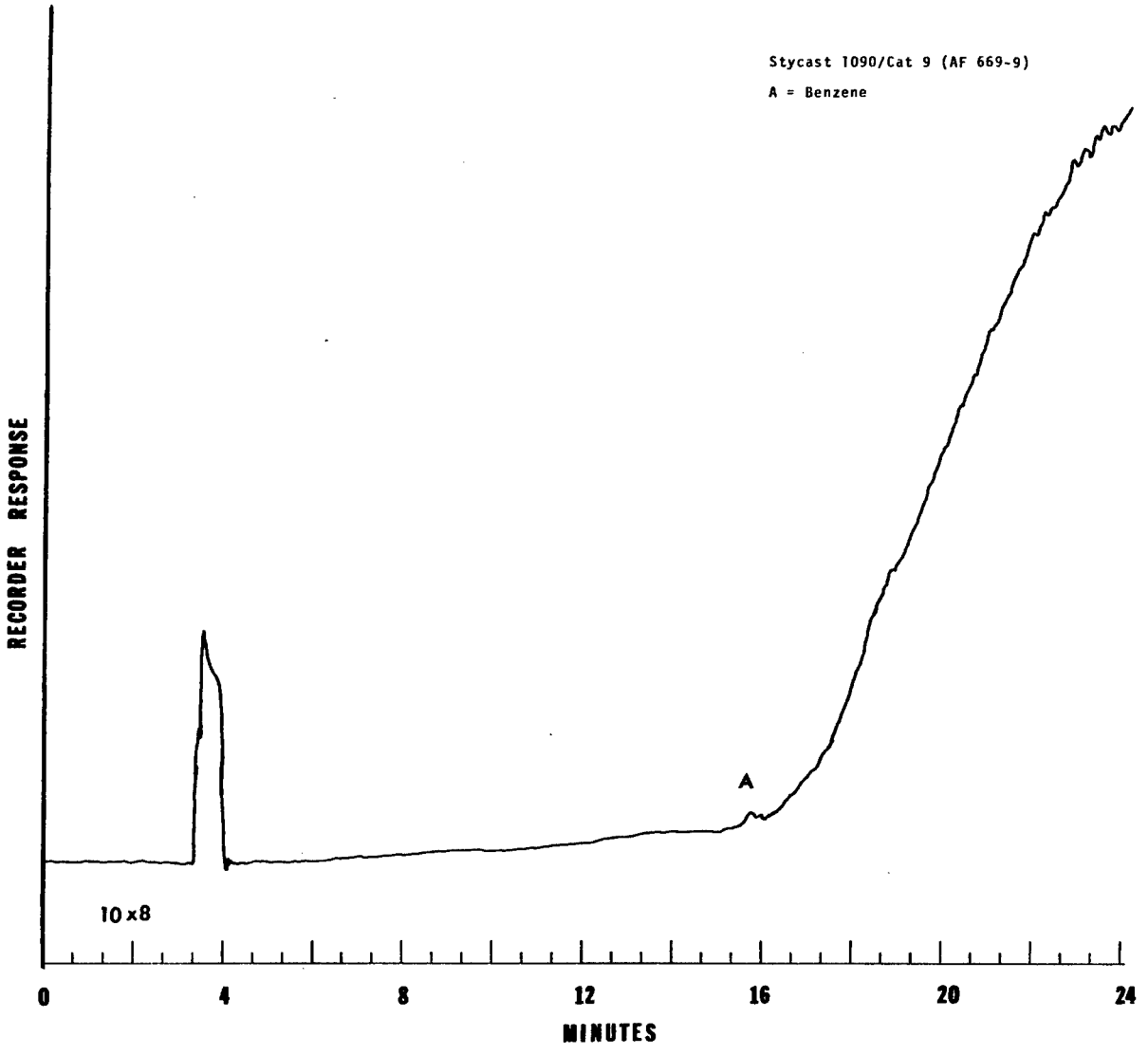

Figure 85. Gas Chromatogram of Gas-Off Products From Stycast 1090/Cat 9 (AF 669-9) (72 Hours @ 68°C).

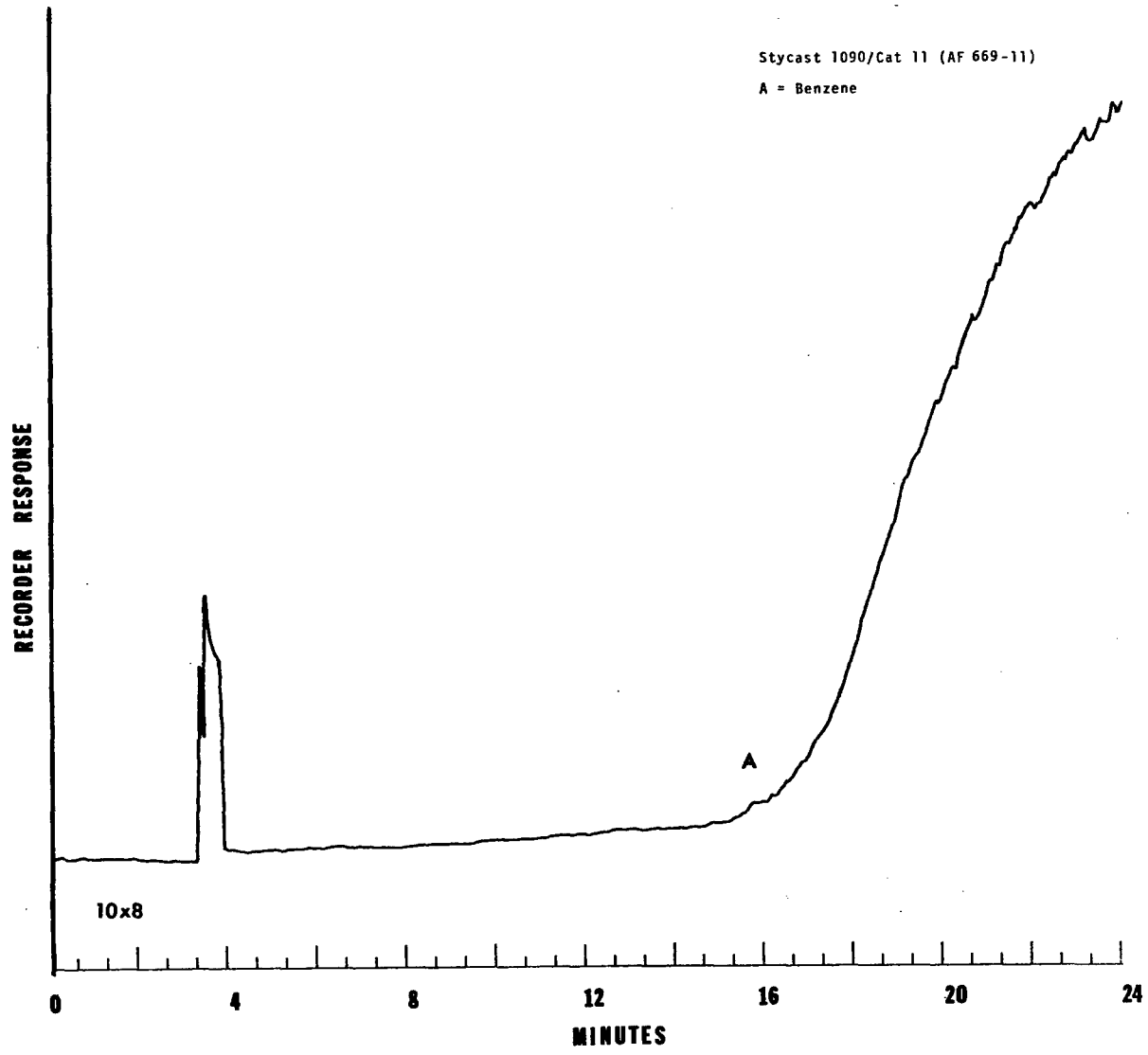

Figure 86. Gas Chromatogram of Gas-Off Products From Stycast 1090/Cat 11 (AF 669-11) (72 Hours @ 68°C).

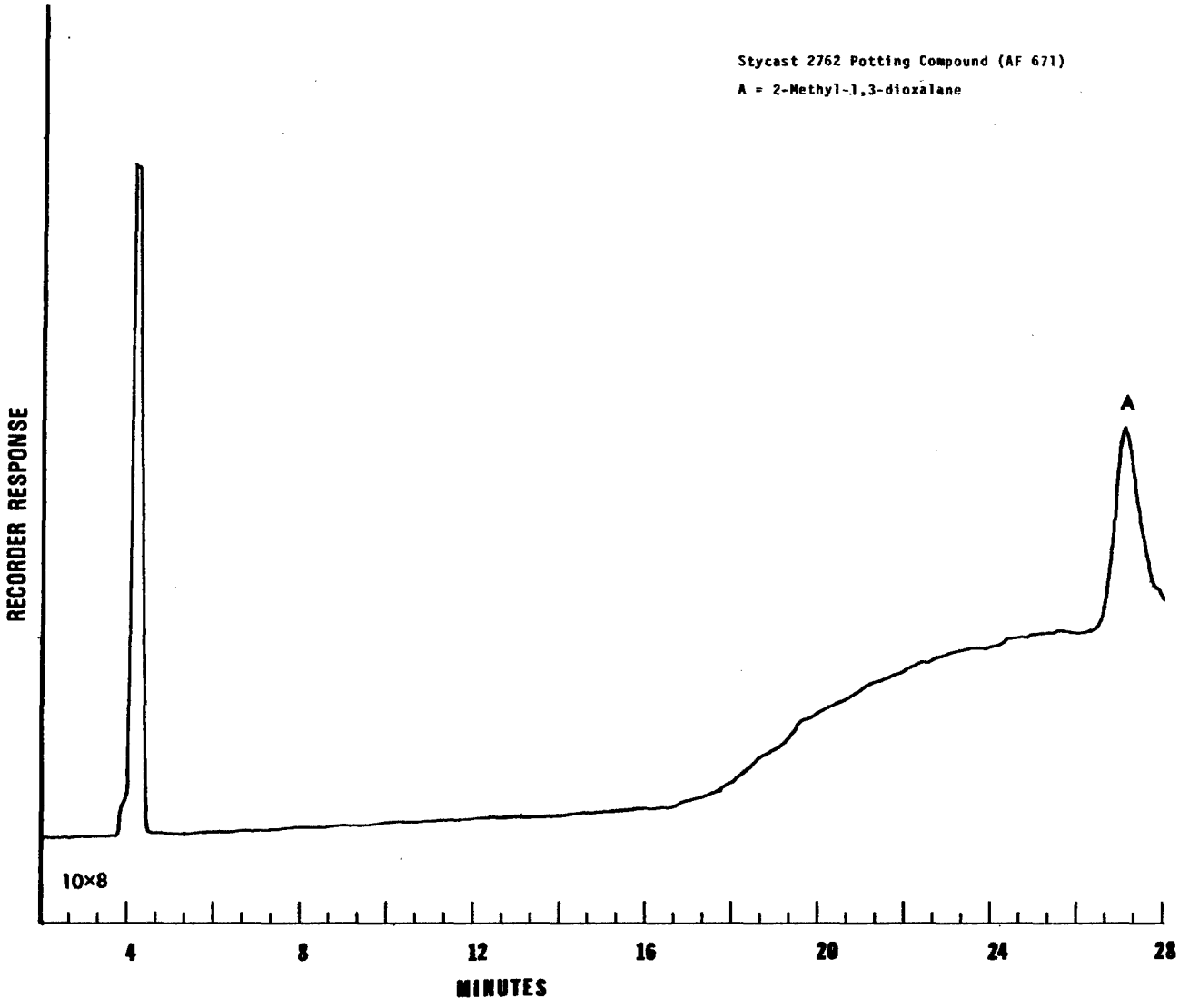

Figure 87. Gas Chromatogram of Gas-Off Products From Stycast 2762 Potting Compound (AF 671) (72 Hours @ 68°C).

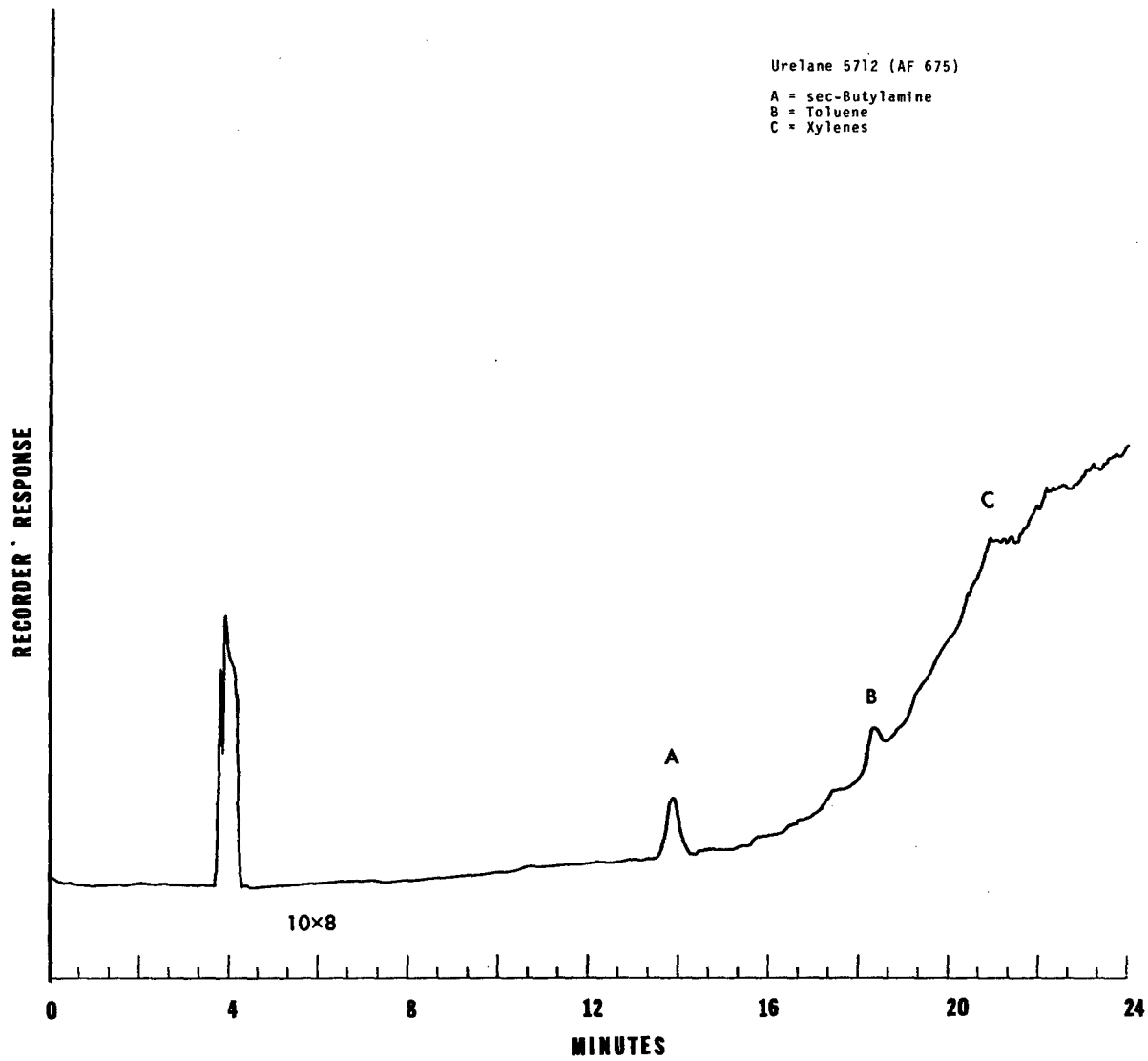

Figure 88. Gas Chromatogram of Gas-Off Products From Urelane 5712 (AF 675) (72 Hours @ 68°C).

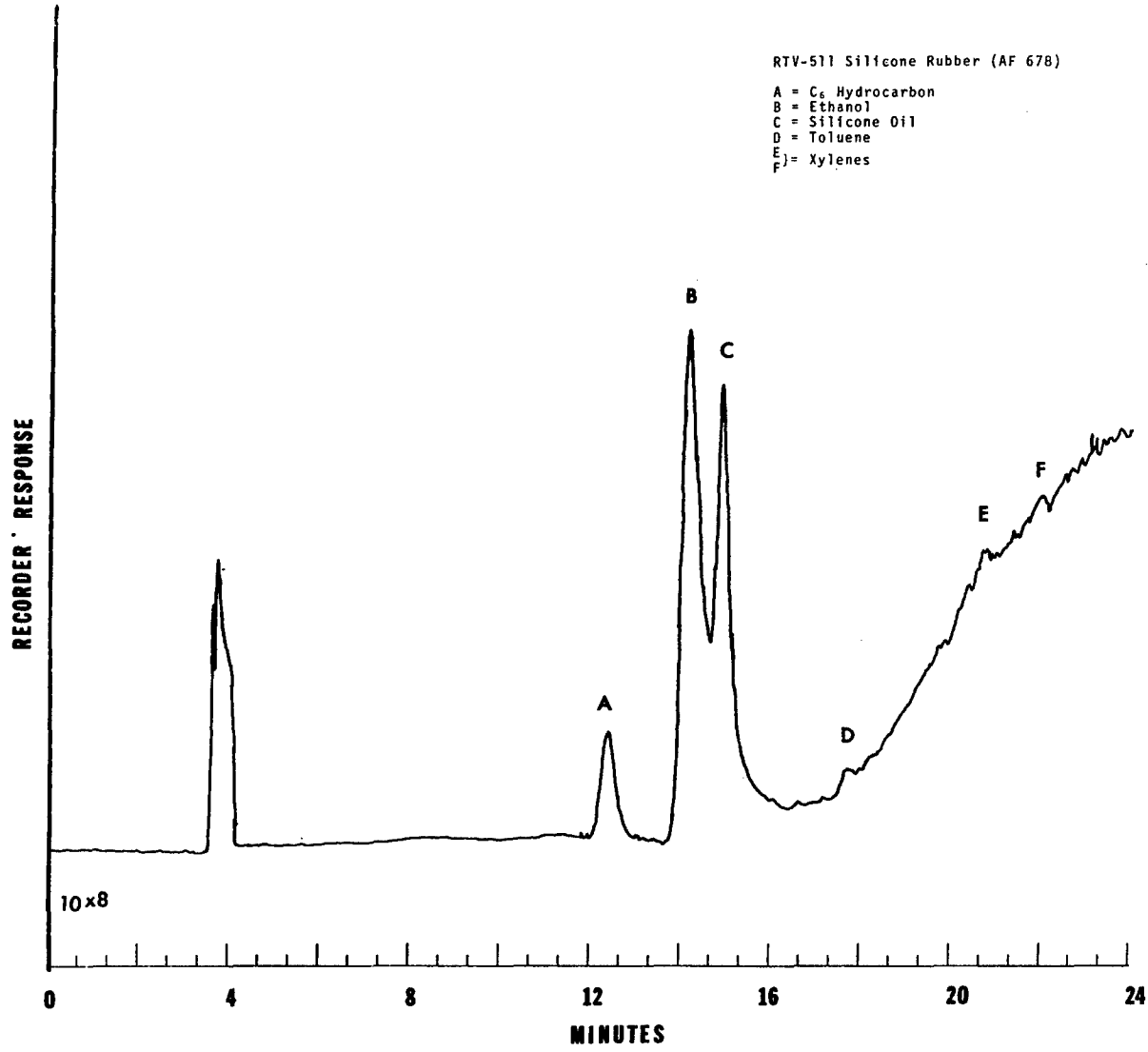

Figure 89. Gas Chromatogram of Gas-Off Products From RTV-511 Silicone Rubber (AF 678) (72 Hours @ 68°C).

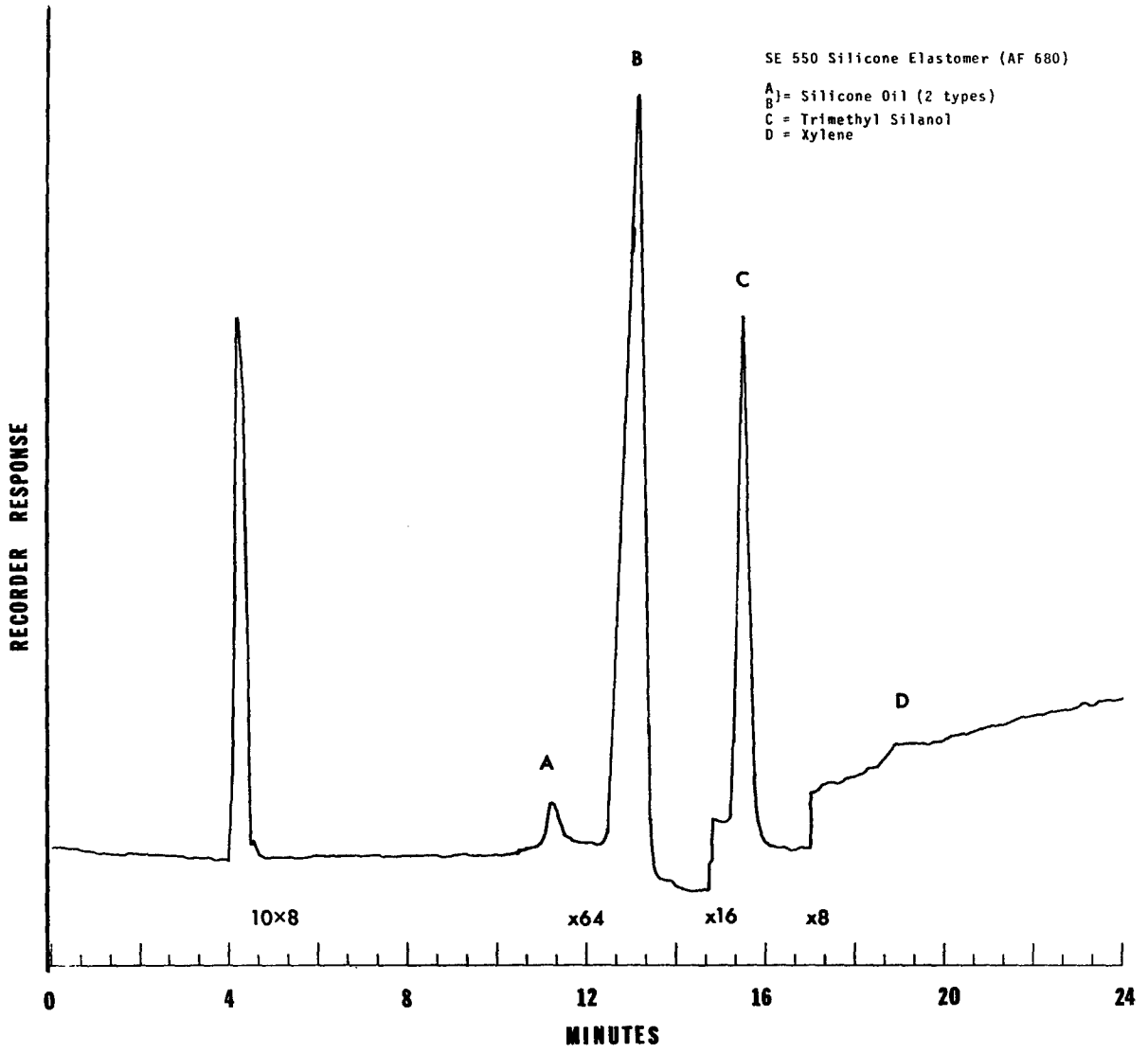

Figure 90. Gas Chromatogram of Gas-Off Products From SE 550 Silicone Elastomer (AF 680) (30 Days @ 25°C).

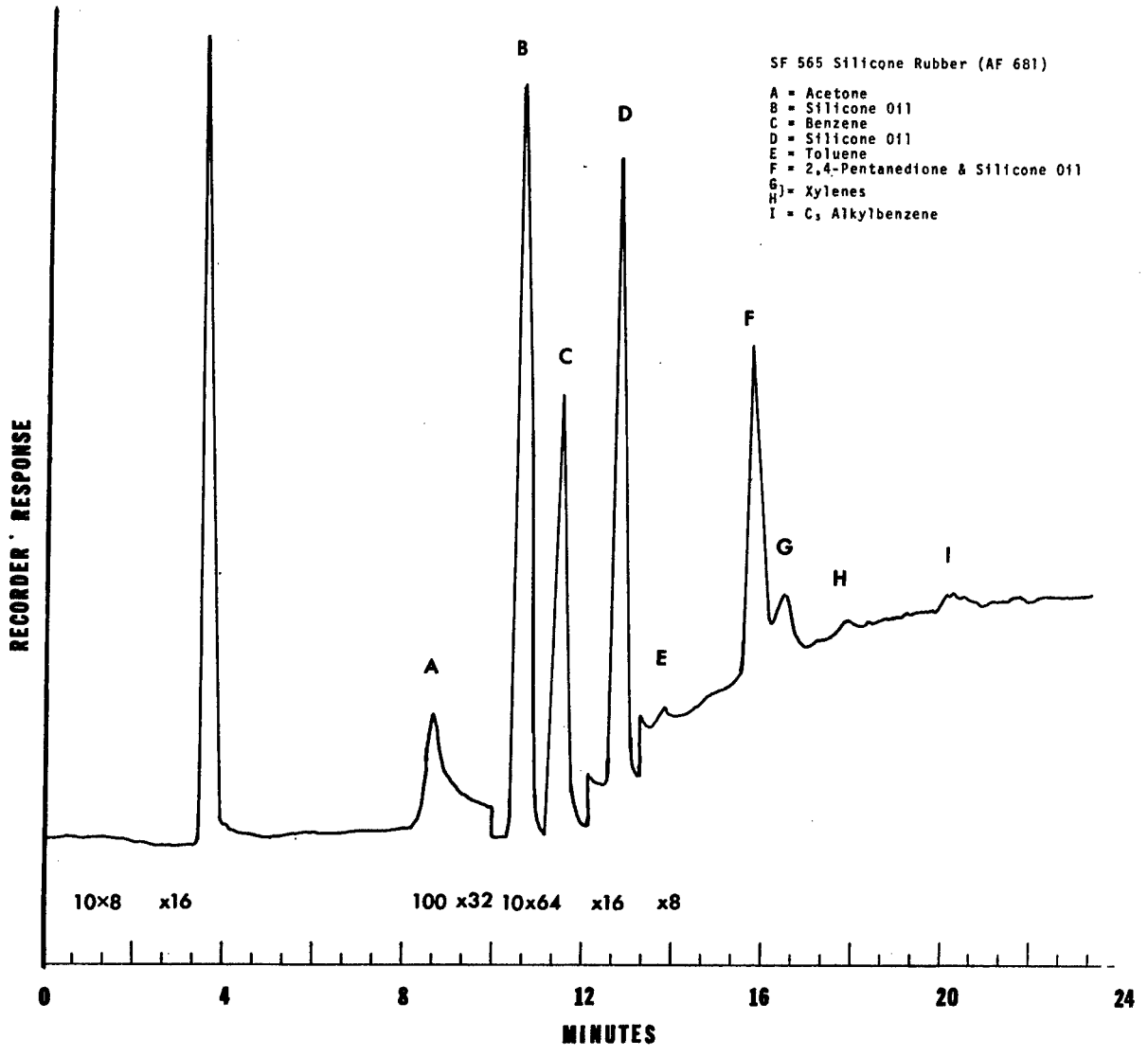

Figure 91. Gas Chromatogram of Gas-Off Products From SF 565 Silicone Rubber (AF 681) (72 Hours @ 68°C).

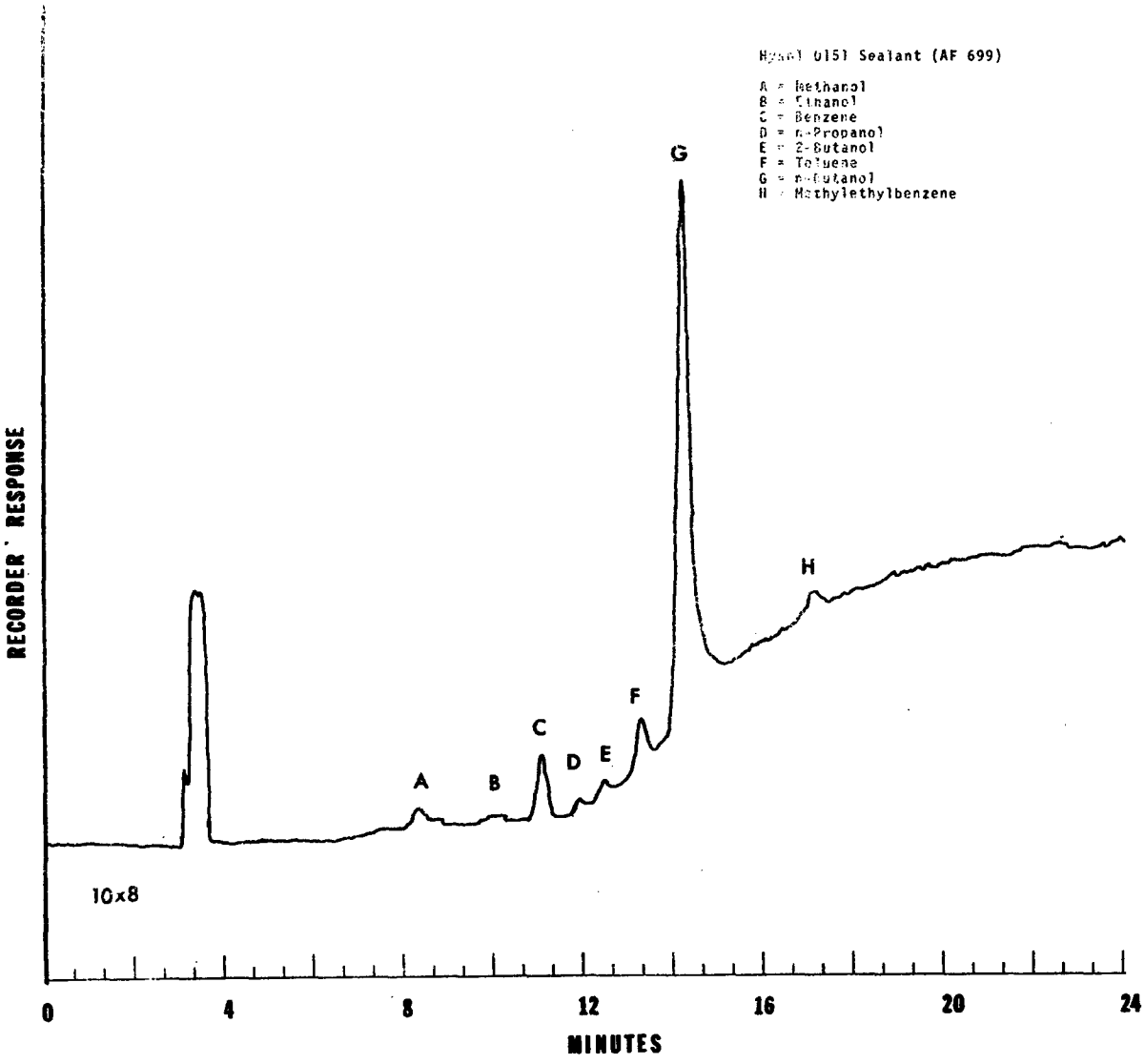

Figure 92. Gas Chromatogram of Gas-Off Products From Hysol 0151 Sealant (AF 699) (72 Hours @ 68°C).

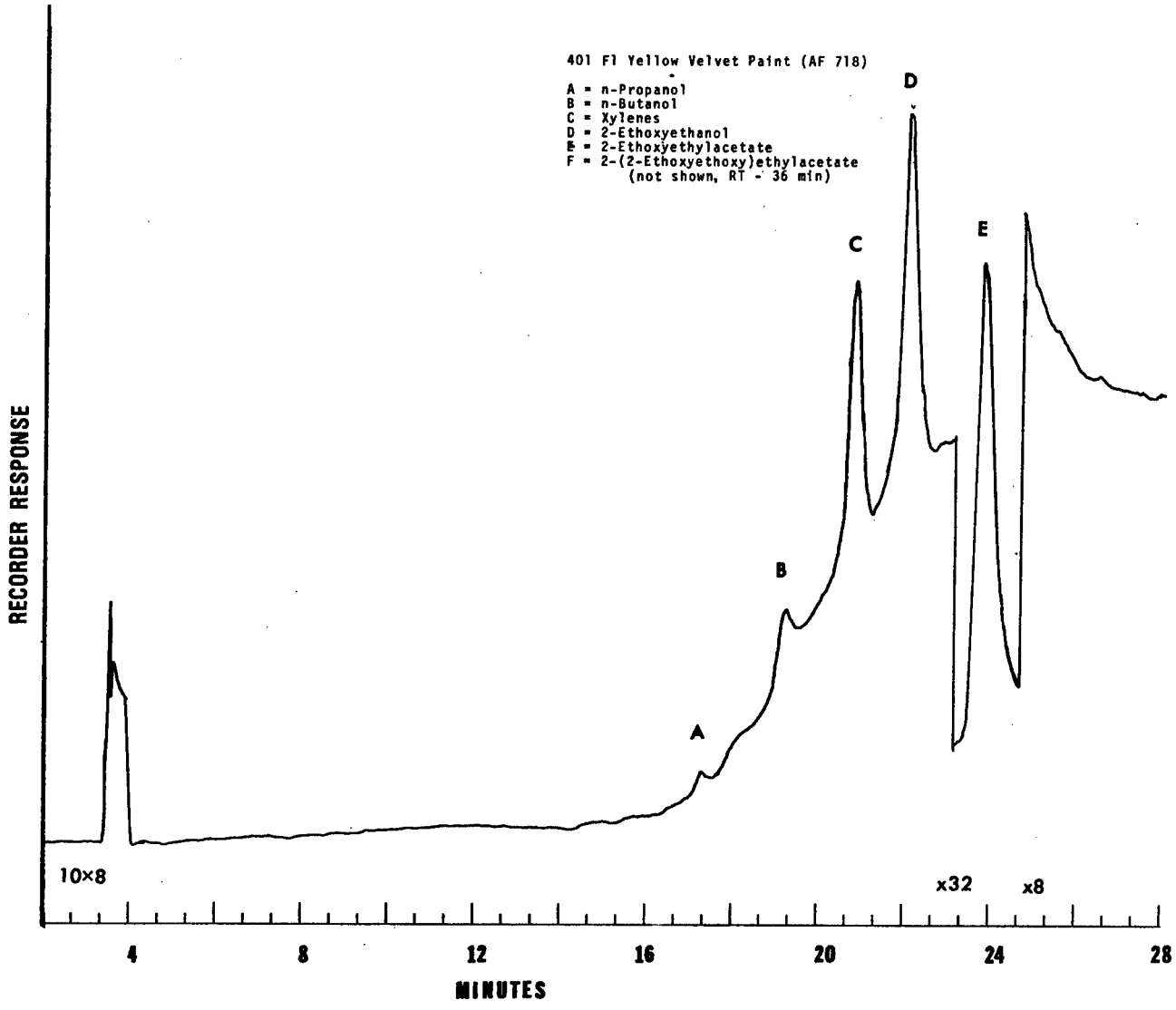

Figure 93. Gas Chromatogram of Gas-Off Products From 401-F1 Yellow Velvet Paint (AF 718) (72 Hours @ 68°C).

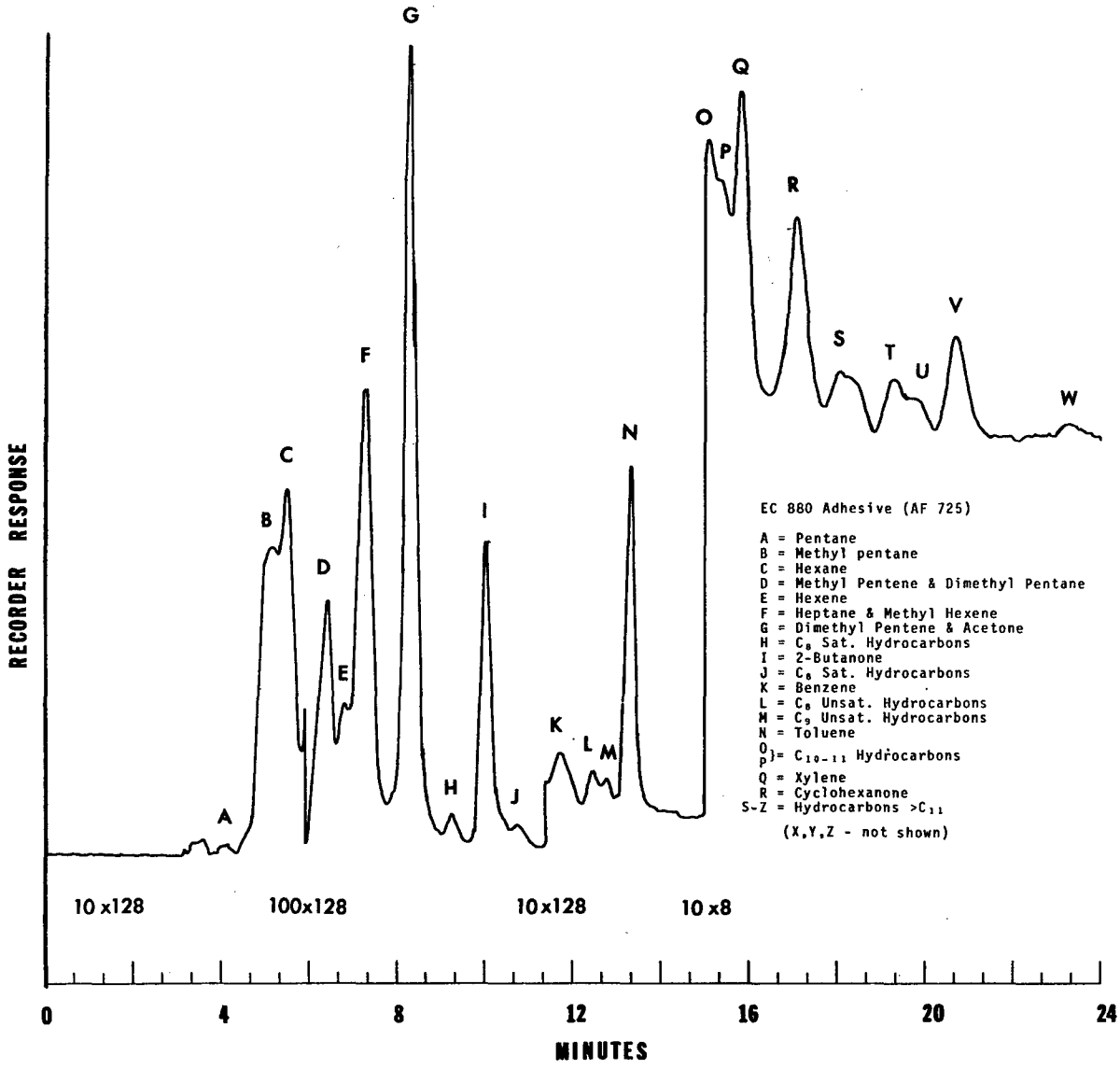

Figure 94. Gas Chromatogram of Gas-Off Products From EC 880 Adhesive (AF 725) (72 Hours @ 68°C).

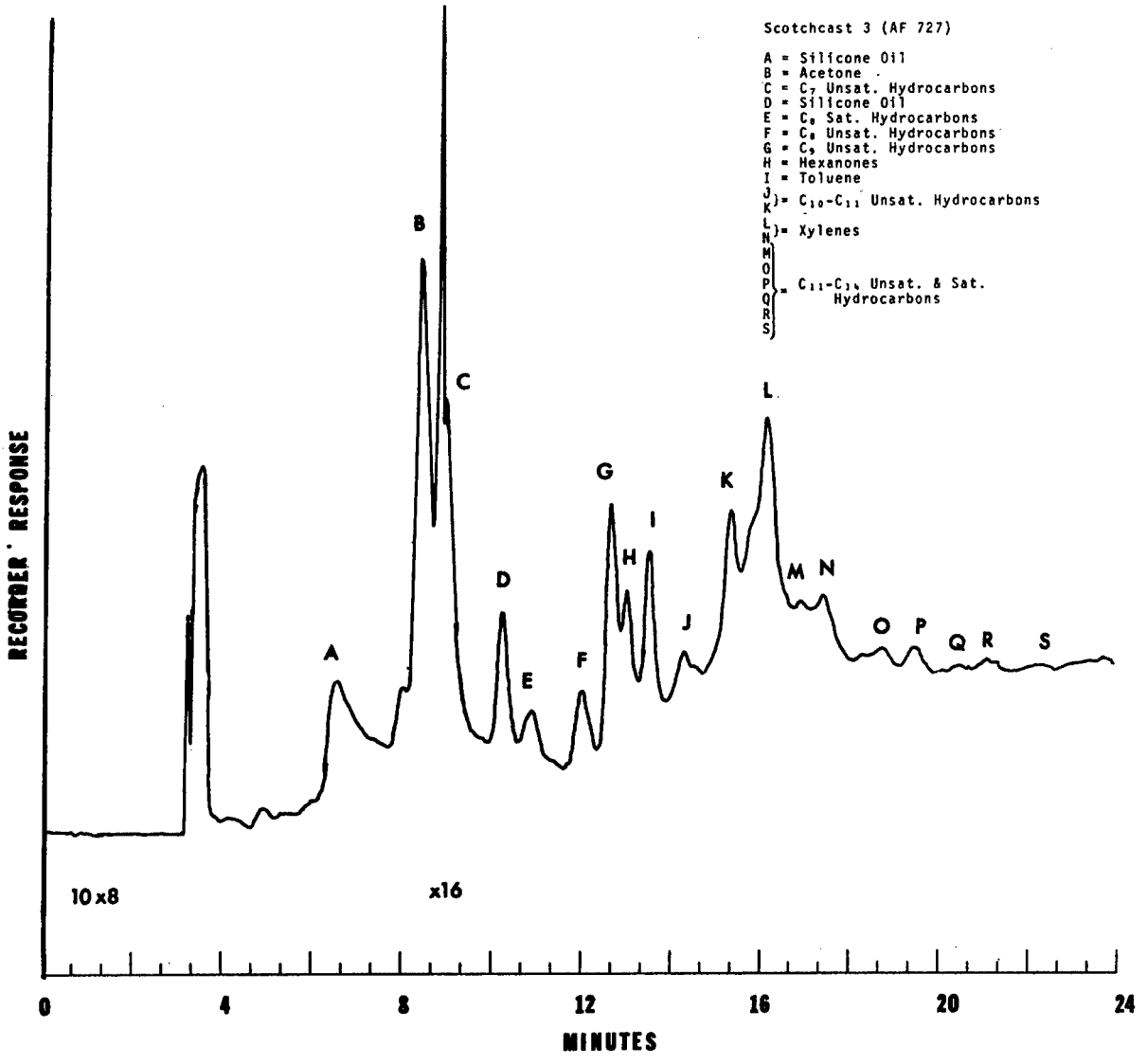

Figure 95. Gas Chromatogram of Gas-Off Products From Scotchcast 3 (AF 727) (72 Hours @ 68°C).

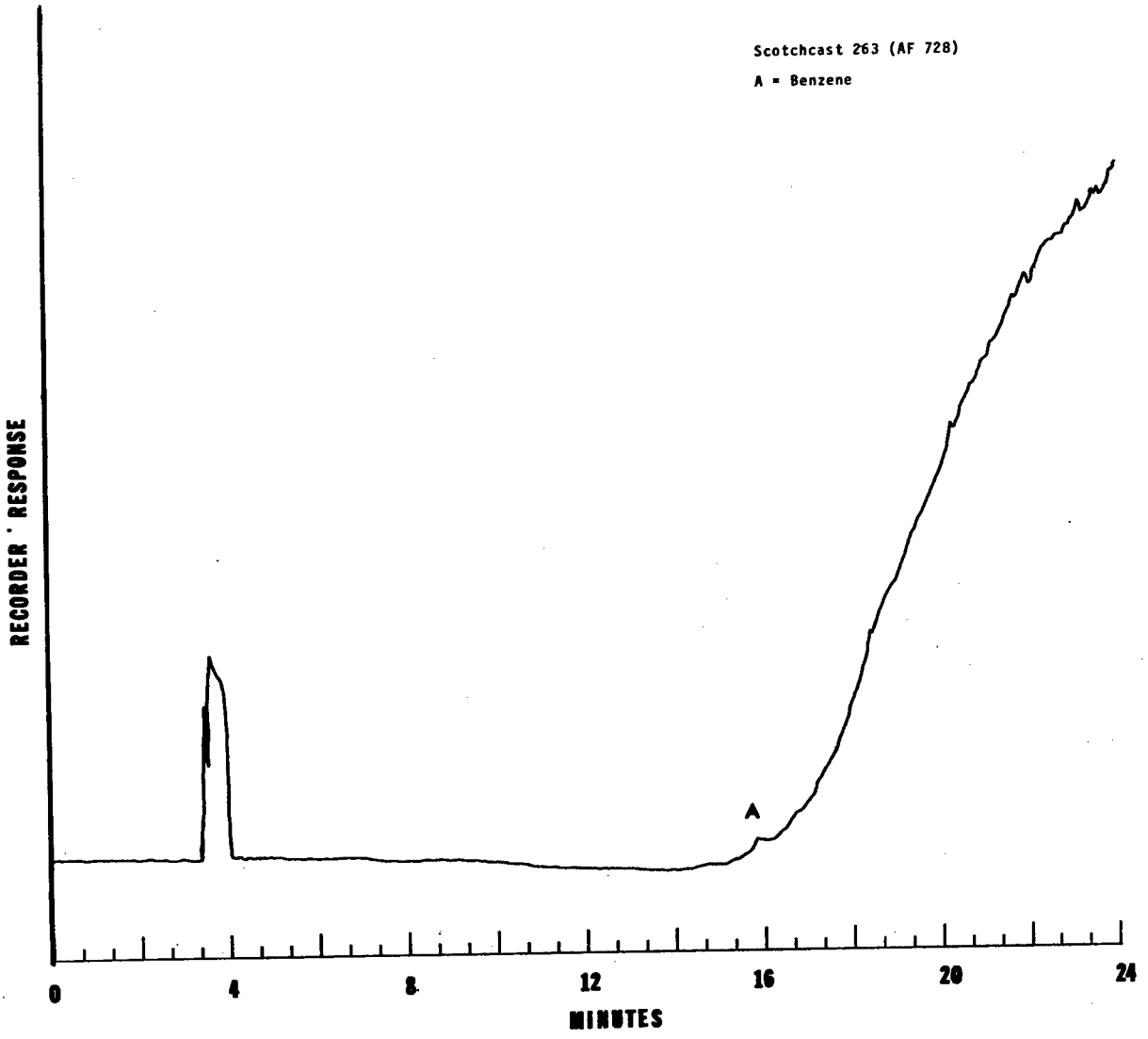

Figure 96. Gas Chromatogram of Gas-Off Products From Scotchcast 263 (AF 728) (72 Hours @ 68°C).

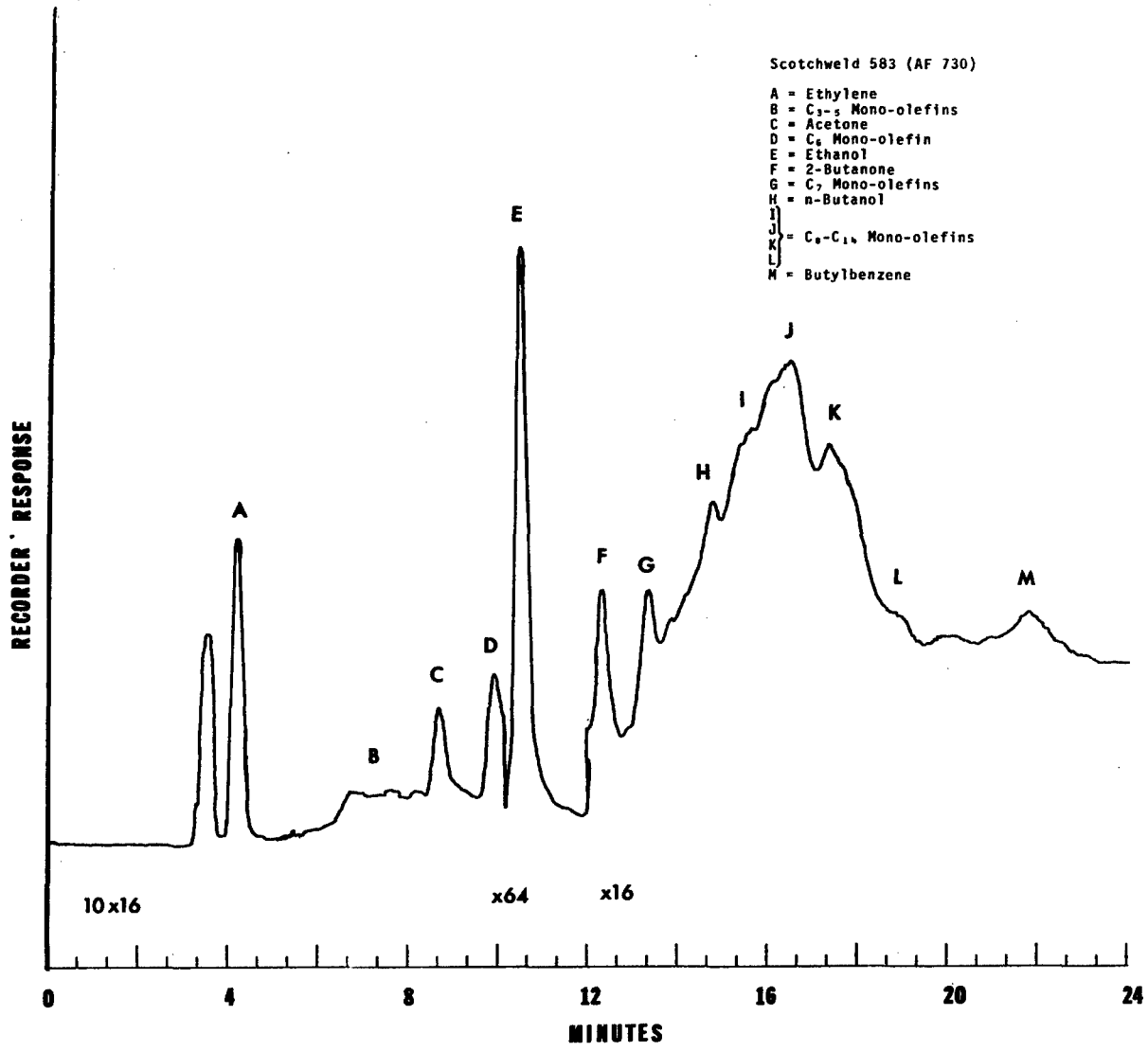

Figure 97. Gas Chromatogram of Gas-Off Products From Scotchweld 583 (AF 730) (72 Hours @ 68°C).

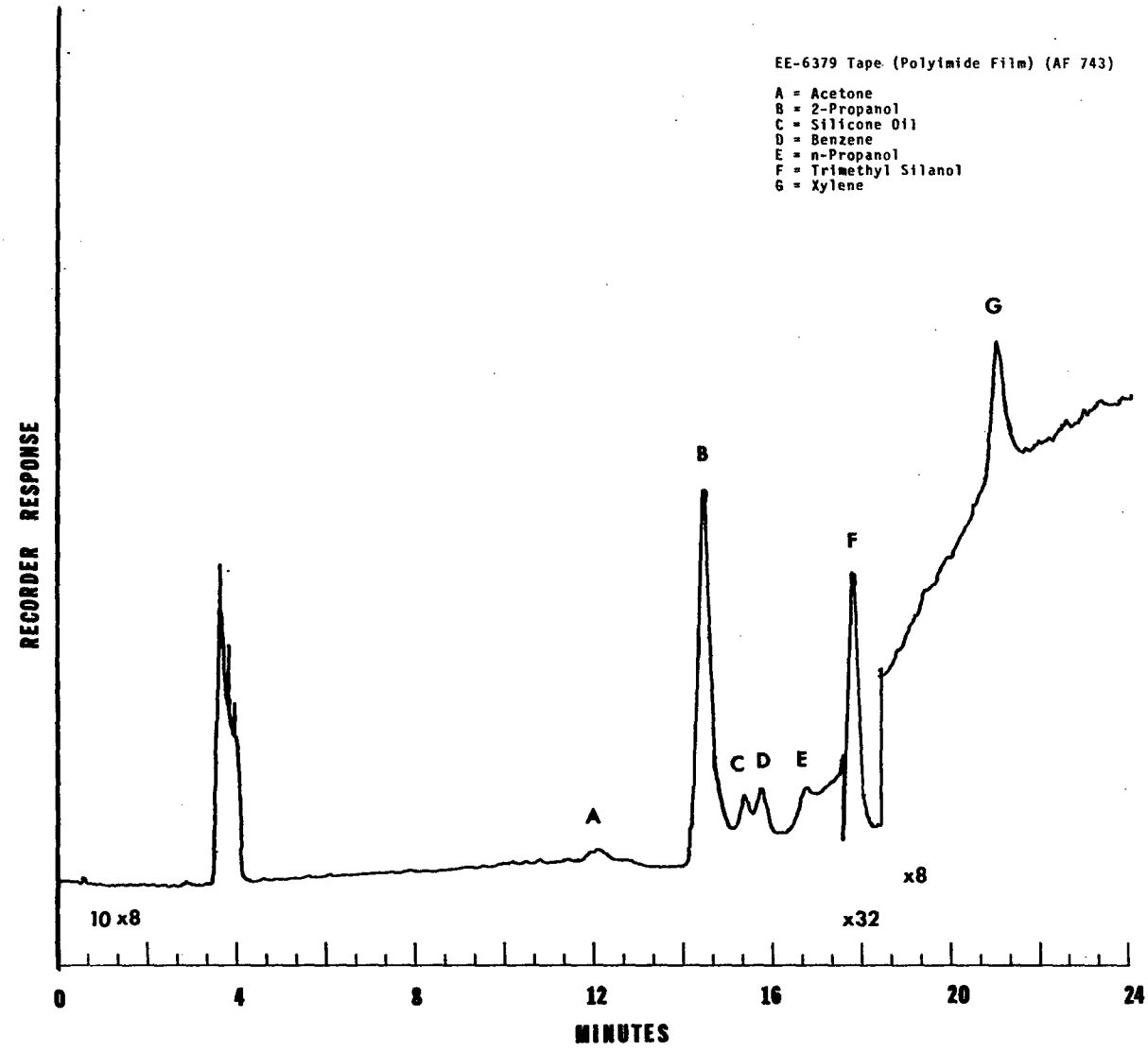

Figure 98. Gas Chromatogram of Gas-Off Products From EE-6379 Tape (Polyimide Film) (AF 743) (72 Hours @ 68°C).

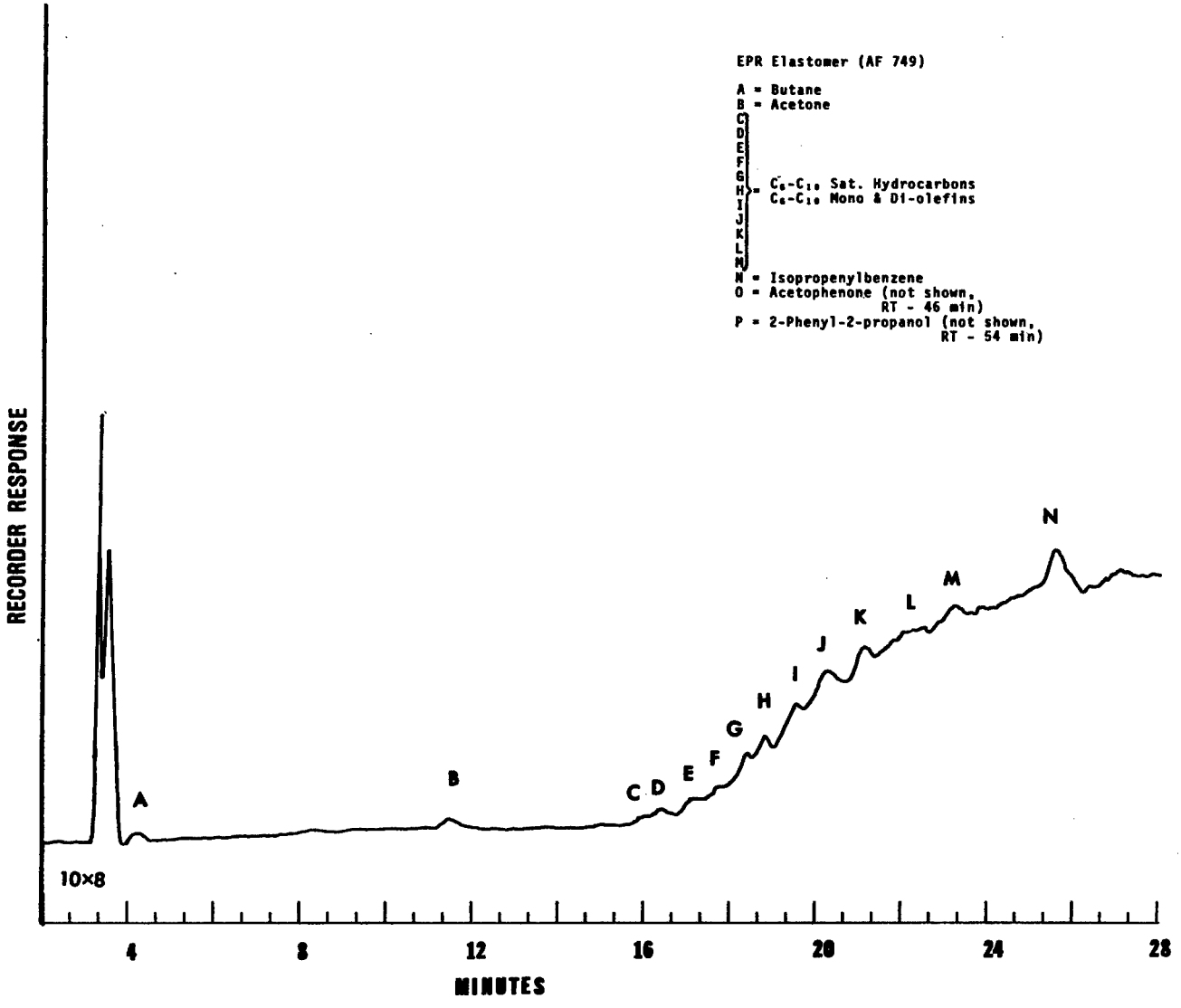

Figure 99. Gas Chromatogram of Gas-Off Products From EPR Elastomer (AF 749) (72 Hours @ 68°C).

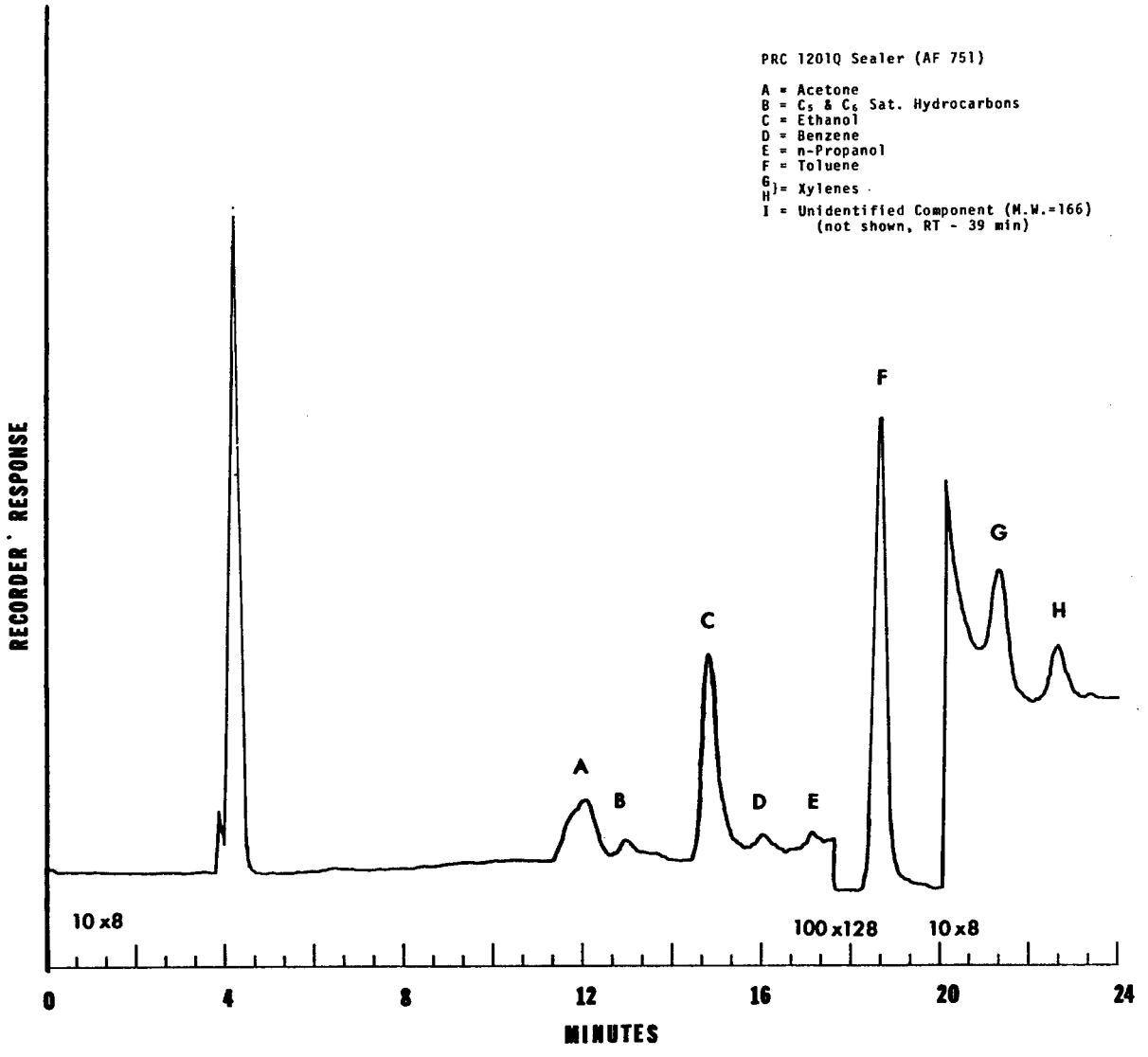

Figure 100. Gas Chromatogram of Gas-Off Products From PRC 1201Q Sealer (AF 751) (72 Hours @ 68°C).

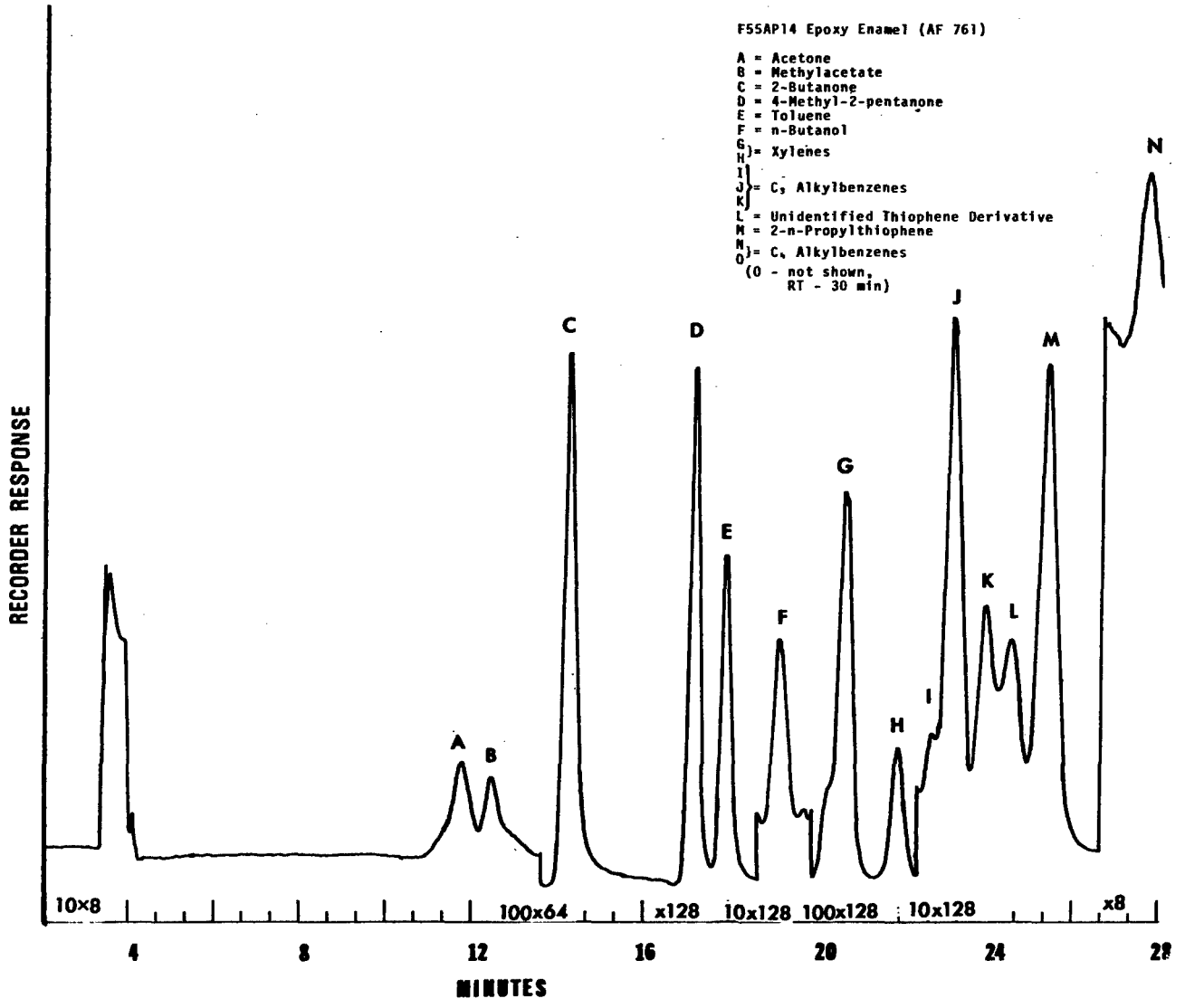

Figure 101. Gas Chromatogram of Gas-Off Products From F55AP14 Epoxy Enamel (AF 761) (72 Hours @ 68°C).

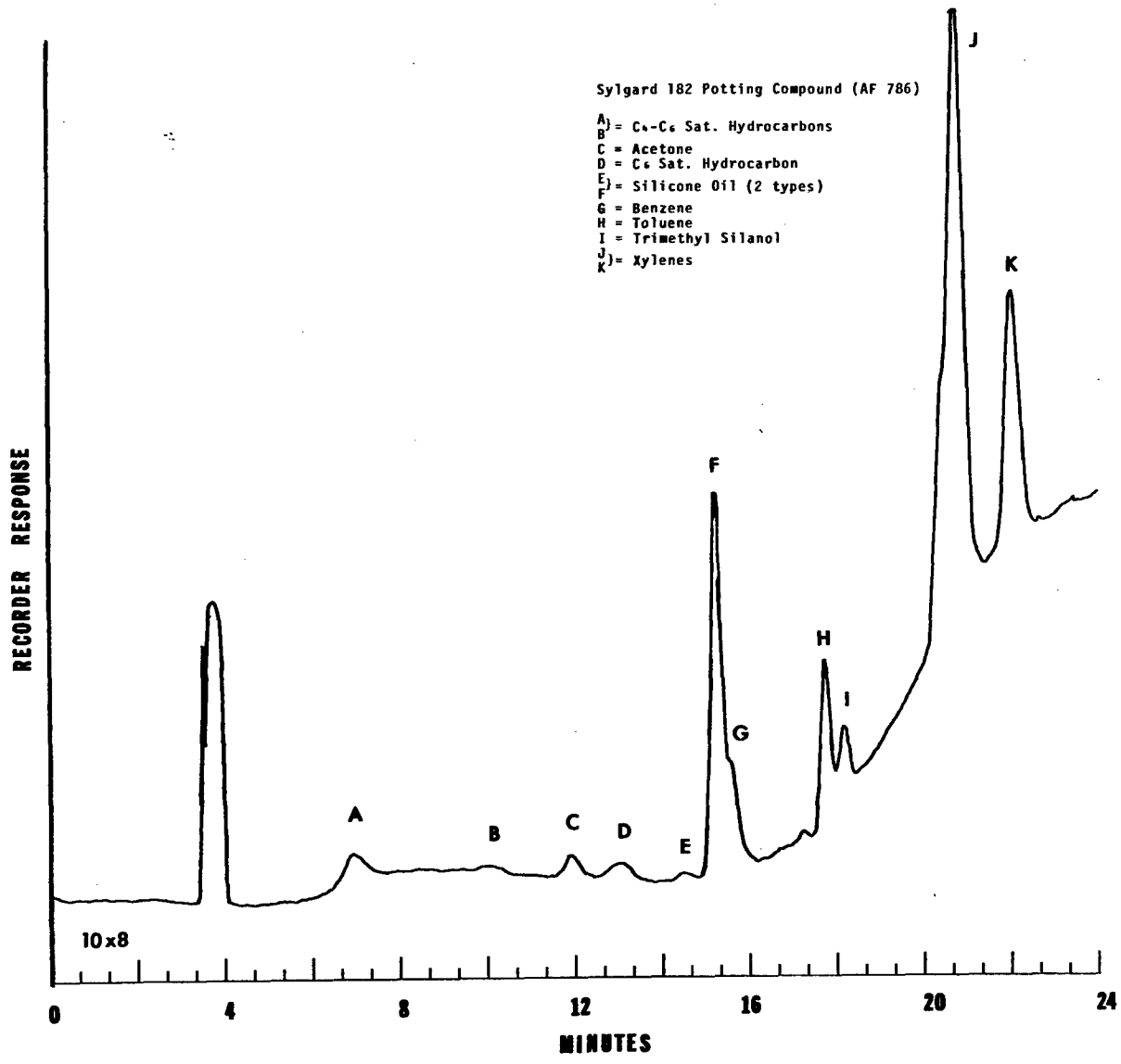

Figure 102. Gas Chromatogram of Gas-Off Products From Sylgard 182 Potting Compound (AF 786) (72 Hours @ 68°C).

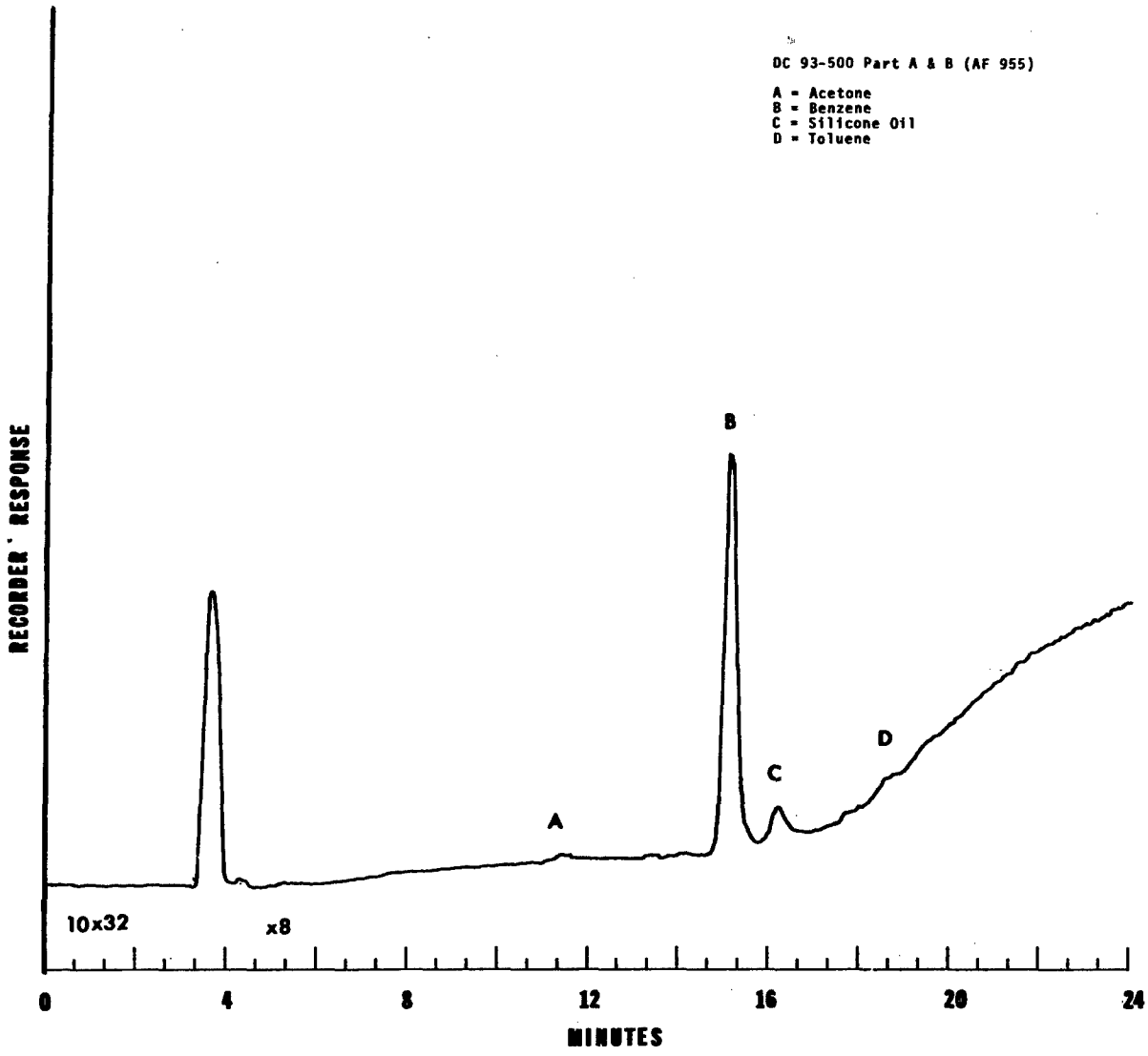

Figure 103. Gas Chromatogram of Gas-Off Products From DC93-500 Part A & B (AF 955) (72 Hours @ 68°C).

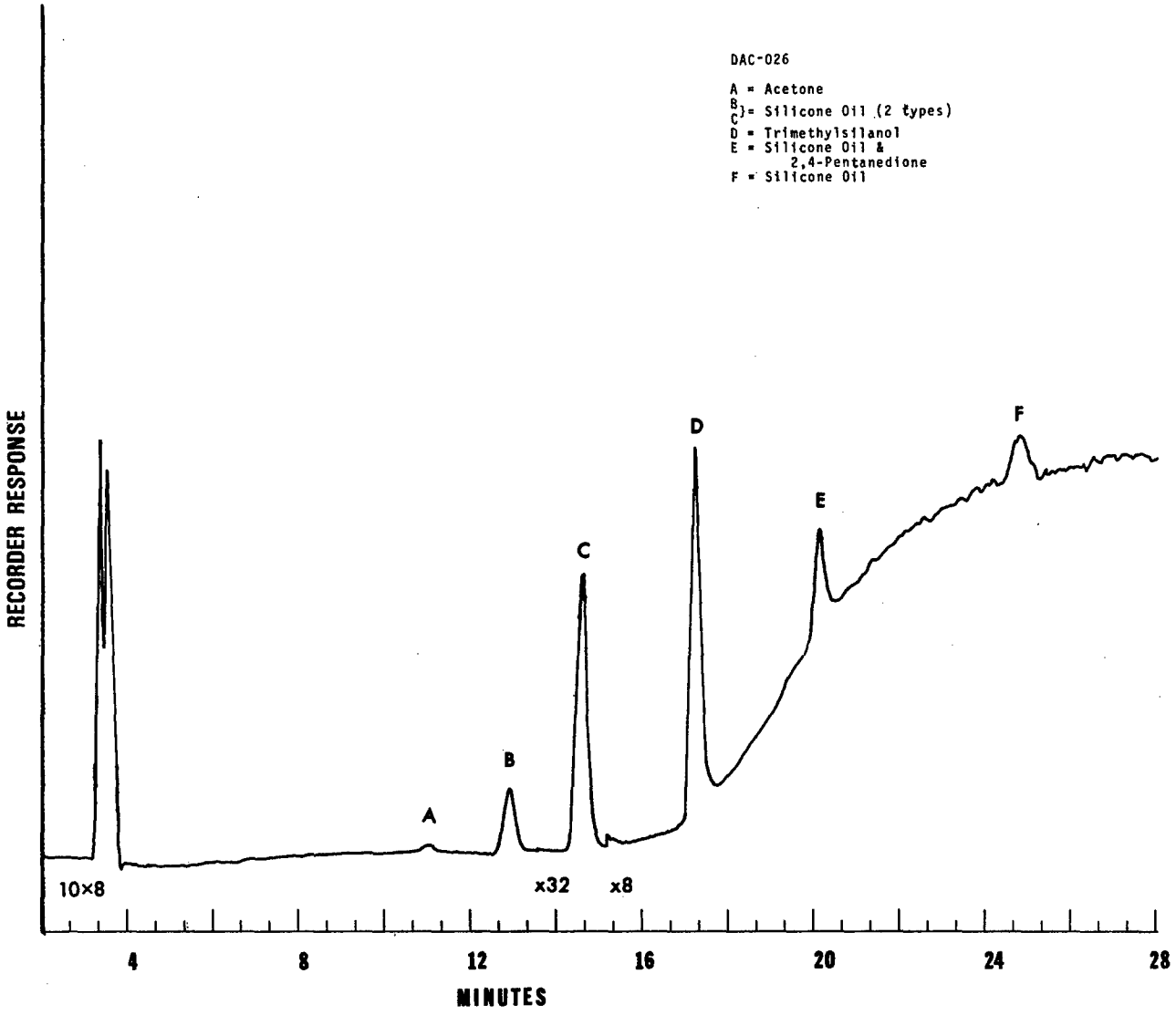

Figure 104. Gas Chromatogram of Gas-Off Products From DAC-026 (72 Hours @ 68°C).

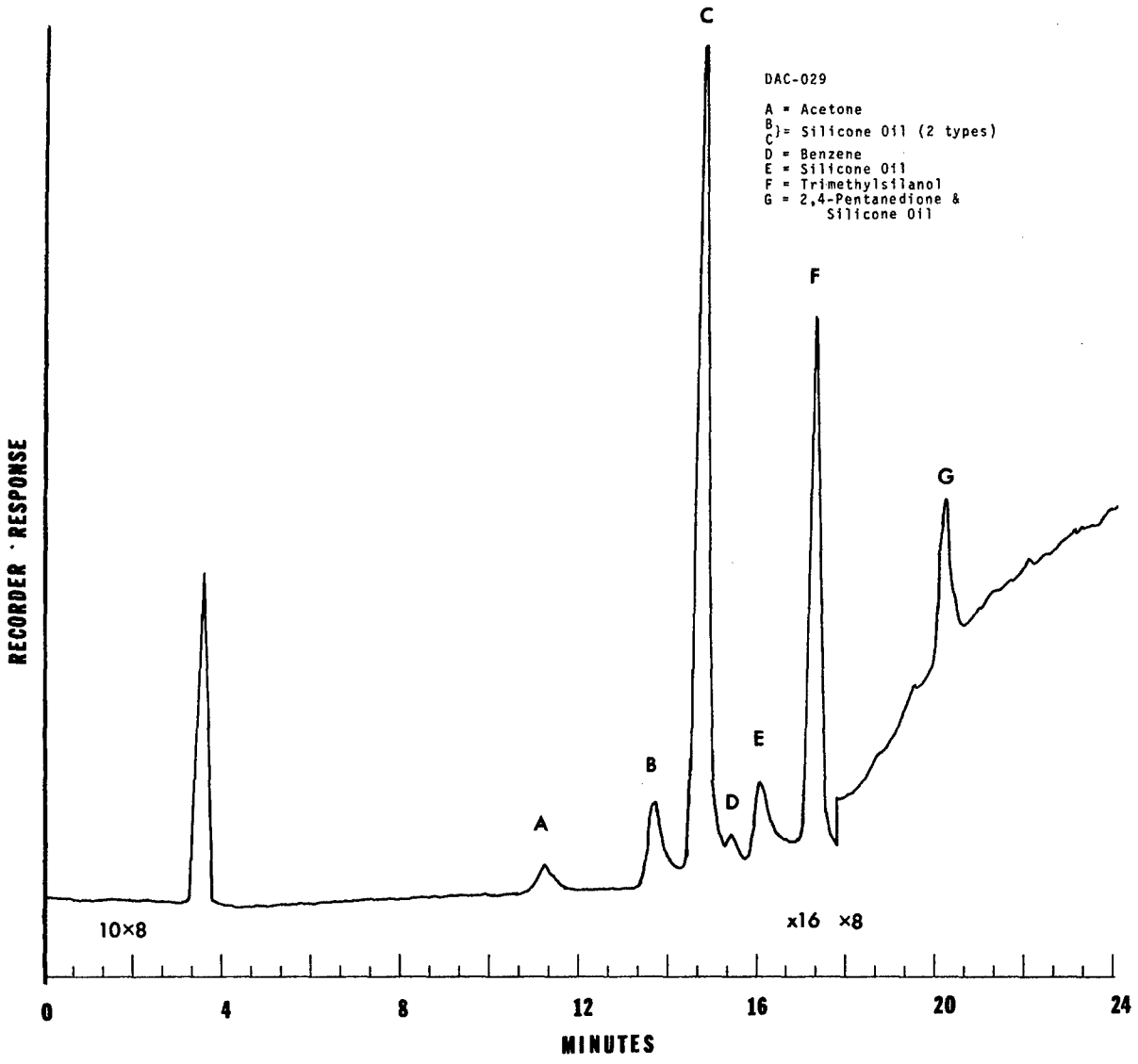

Figure 105. Gas Chromatogram of Gas-Off Products From DAC-029 (72 Hours @ 68°C).

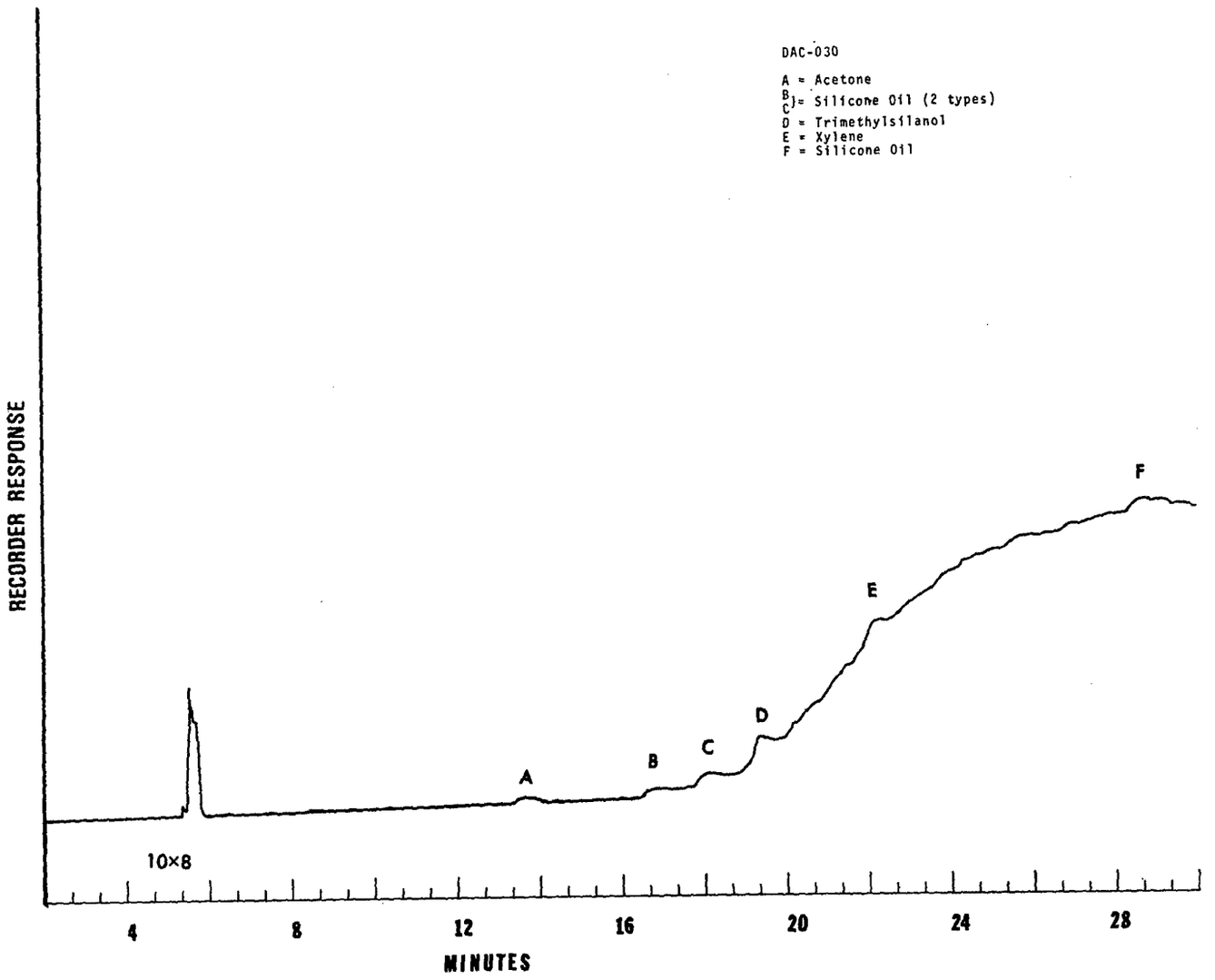

Figure 106. Gas Chromatogram of Gas-Off Products From DAC-030 (72 Hours @ 68°C).

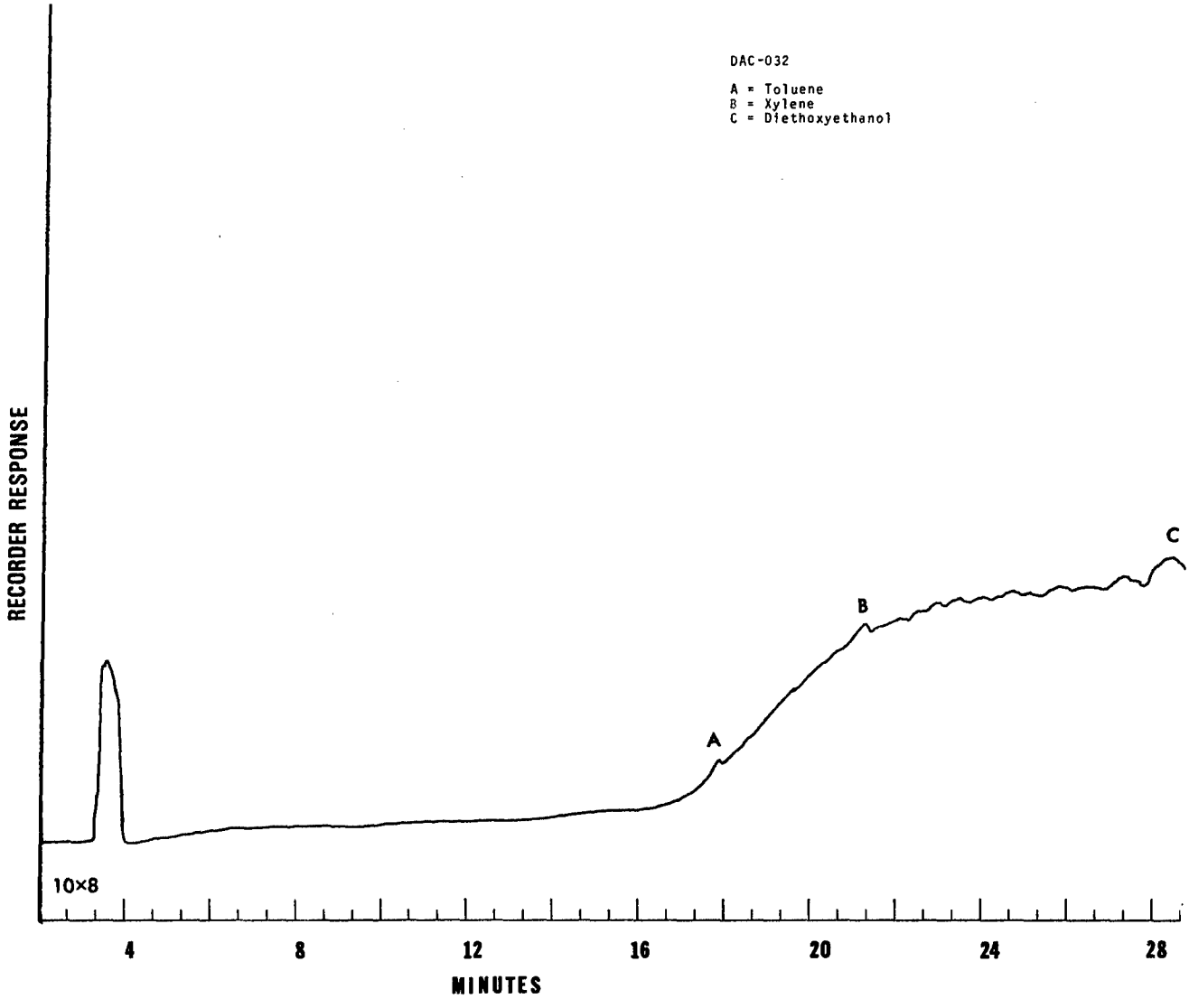

Figure 107. Gas Chromatogram of Gas-Off Products From DAC-032 (72 Hours @ 68°C).

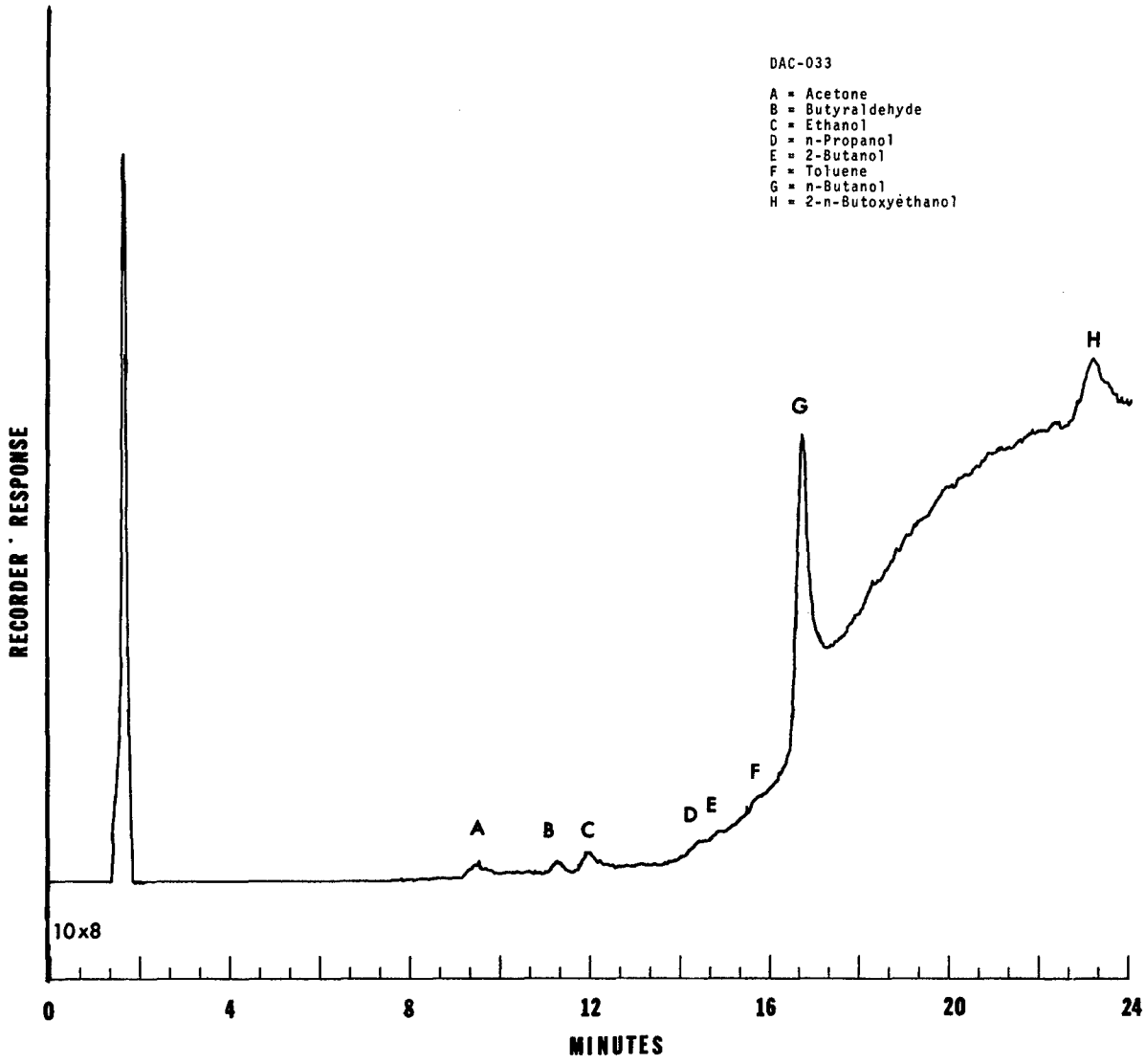

Figure 108. Gas Chromatogram of Gas-Off Products From DAC-033 (72 Hours @ 68°C).

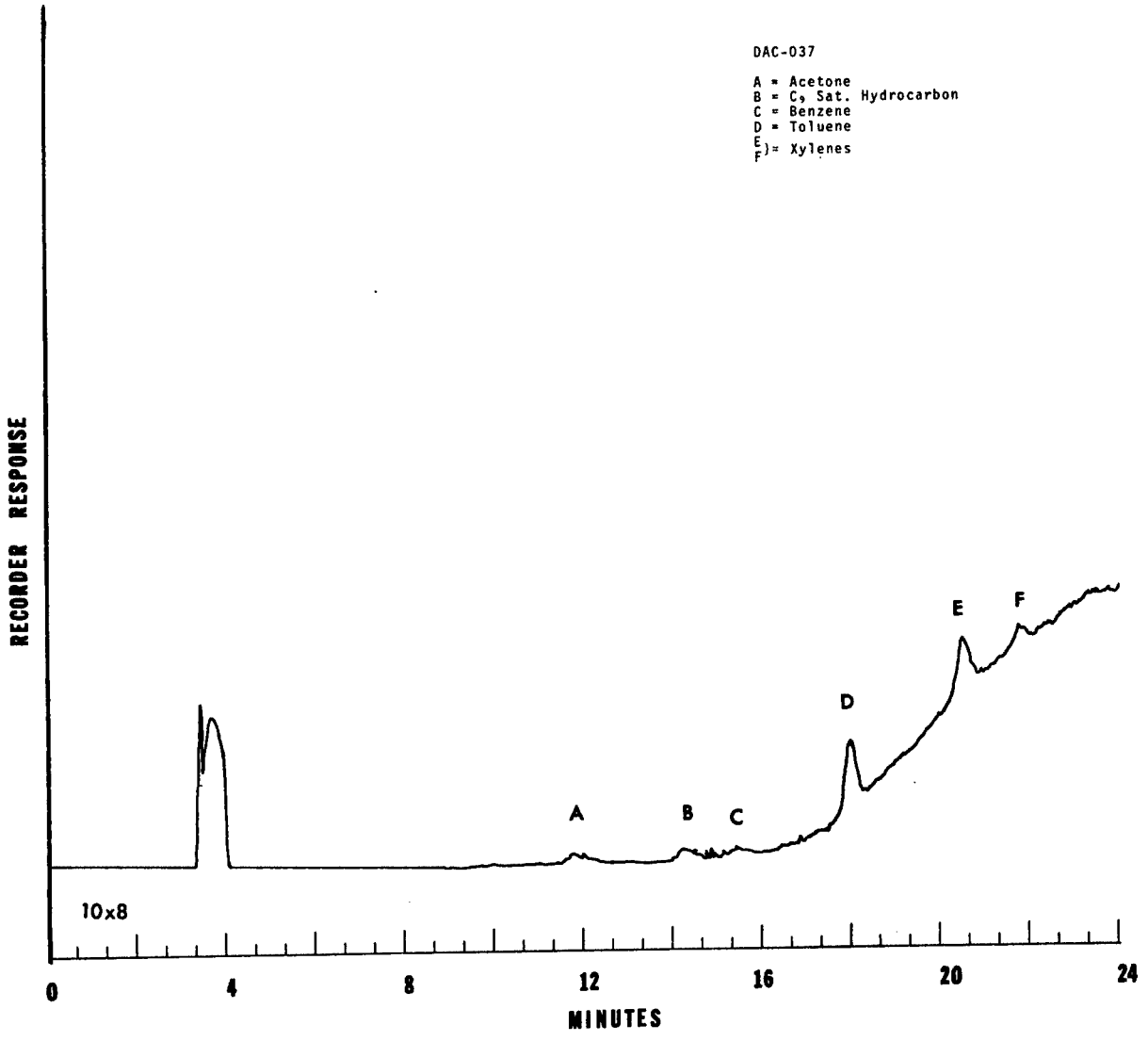

Figure 109. Gas Chromatogram of Gas-Off Products From DAC-037 (72 Hours @ 68°C).

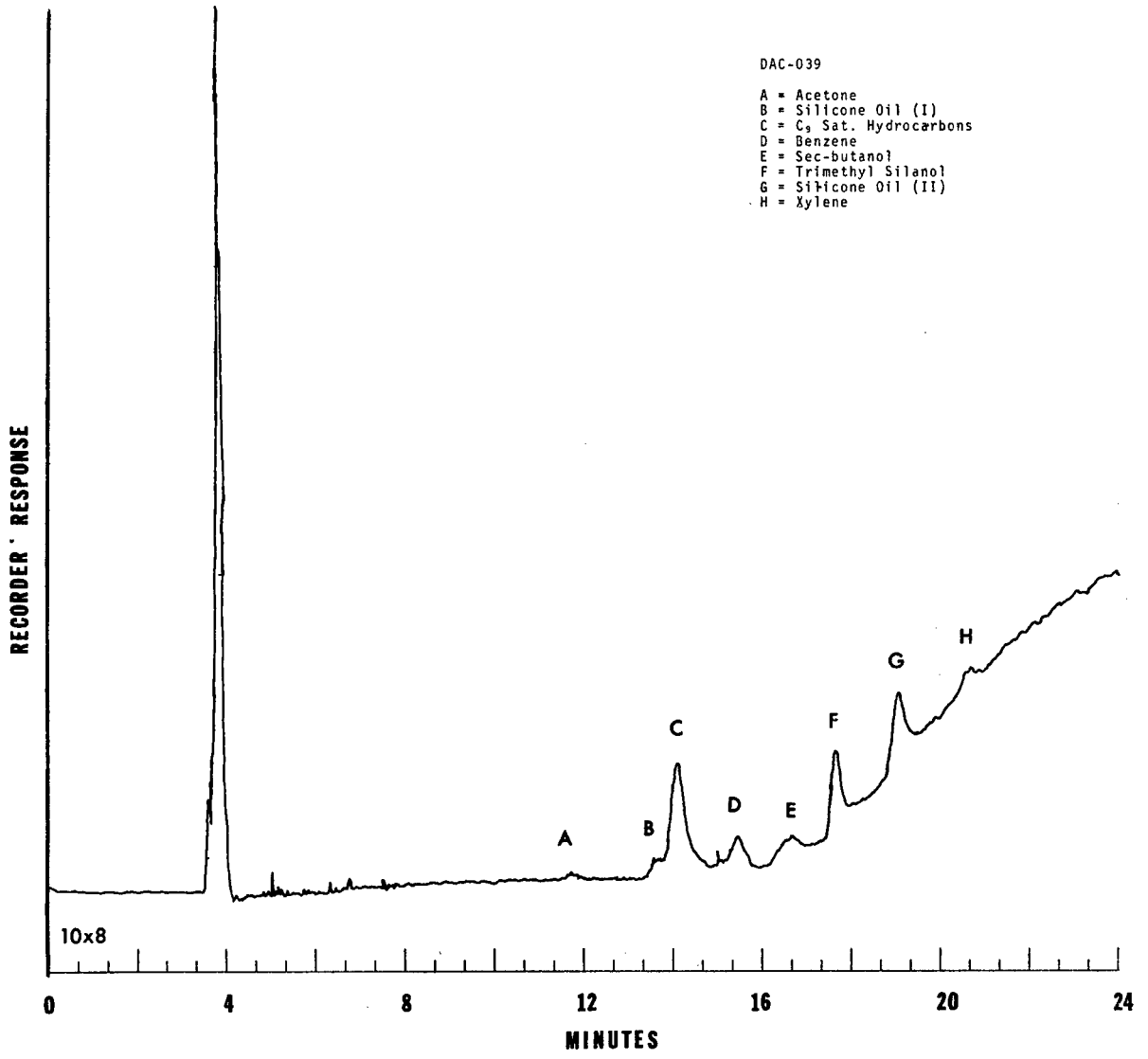

Figure 110. Gas Chromatogram of Gas-Off Products From DAC-039 (72 Hours @ 68°C).

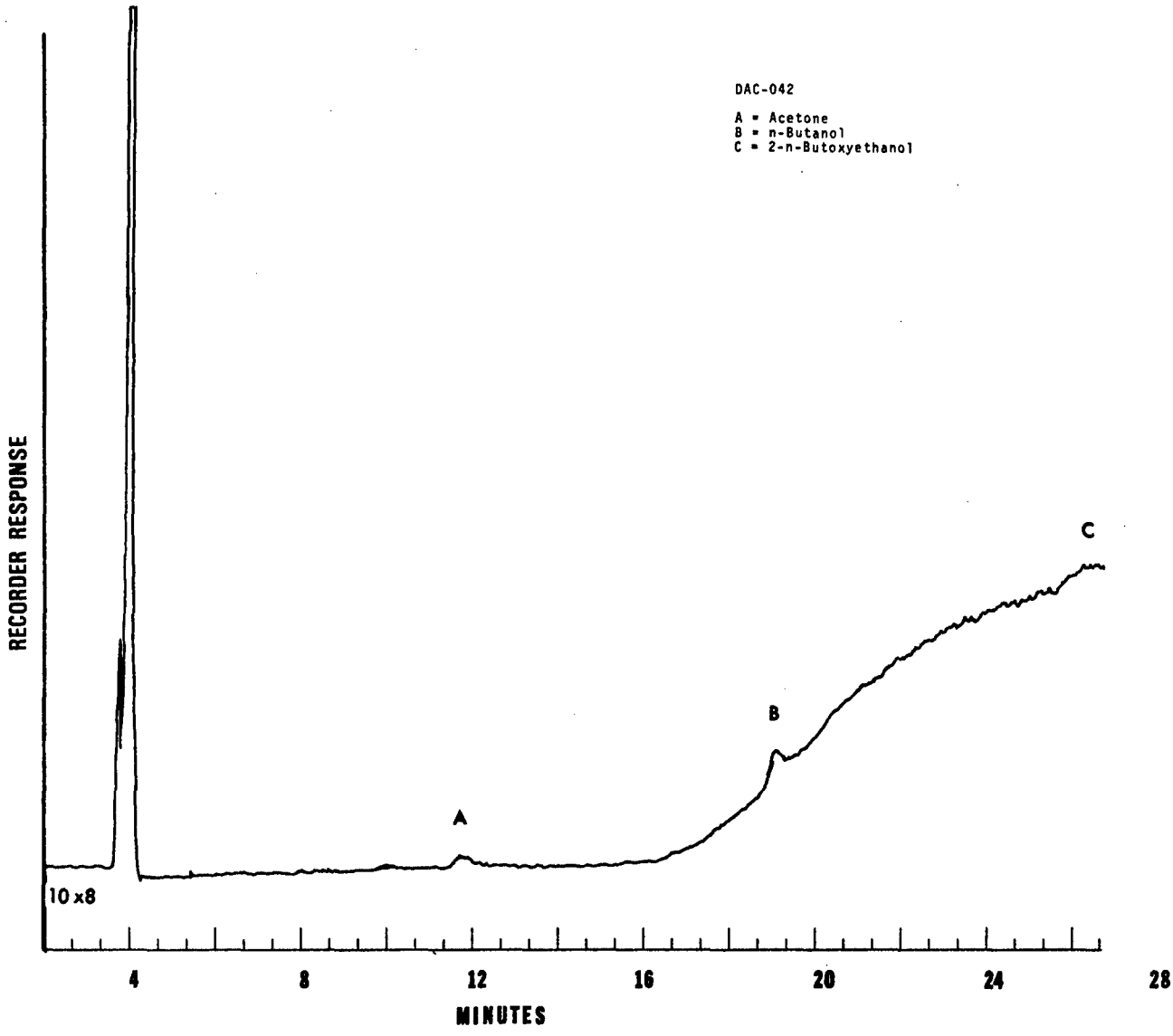

Figure 111. Gas Chromatogram of Gas-Off Products From DAC-042 (72 Hours @ 68°C).

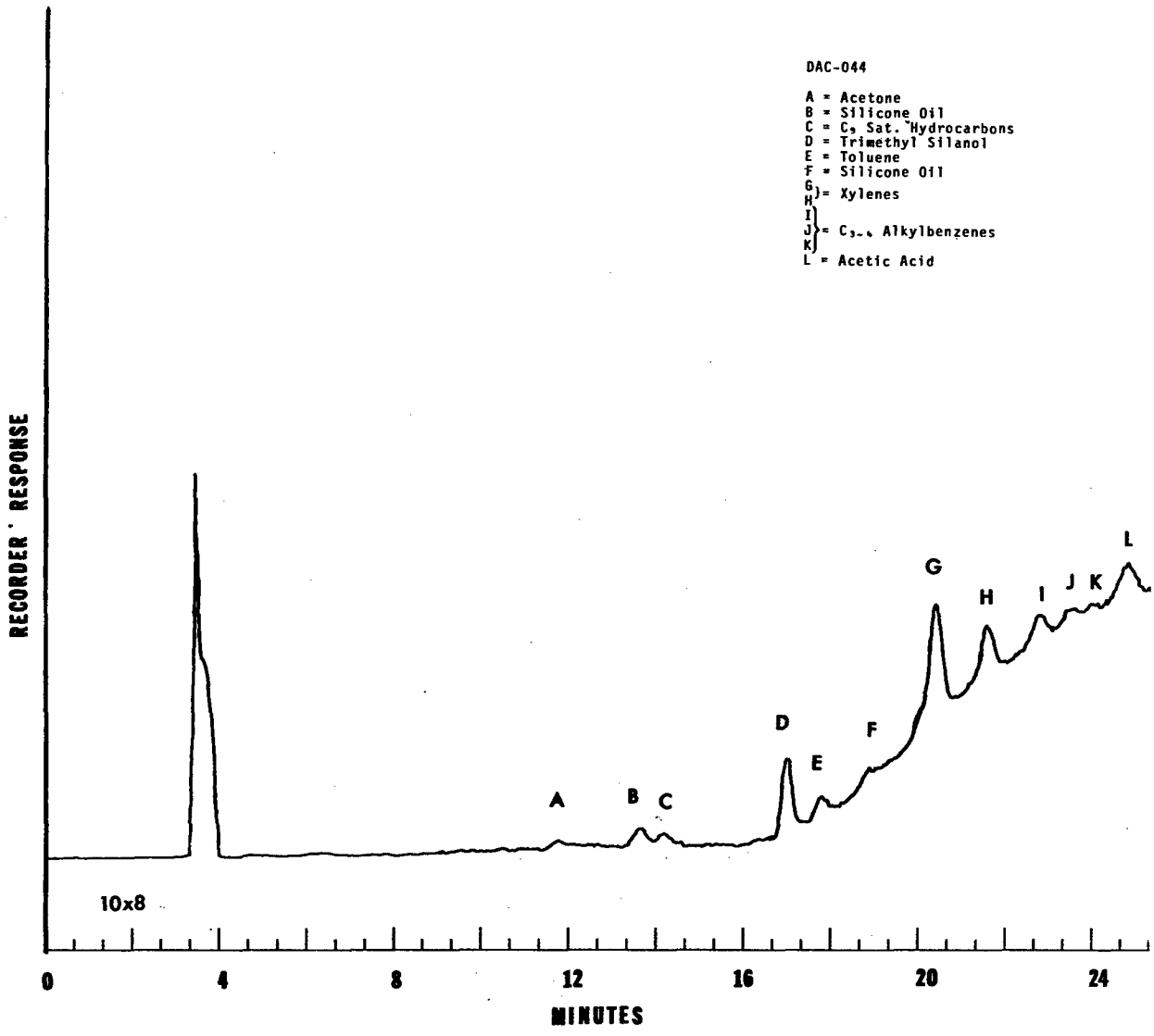

Figure 112. Gas Chromatogram of Gas-Off Products From DAC-044 (72 Hours @ 68°C).

APPENDIX IV

THERMOGRAVIMETRIC (TGA) PATTERNS FOR CARBOXYNITROSO RUBBERS

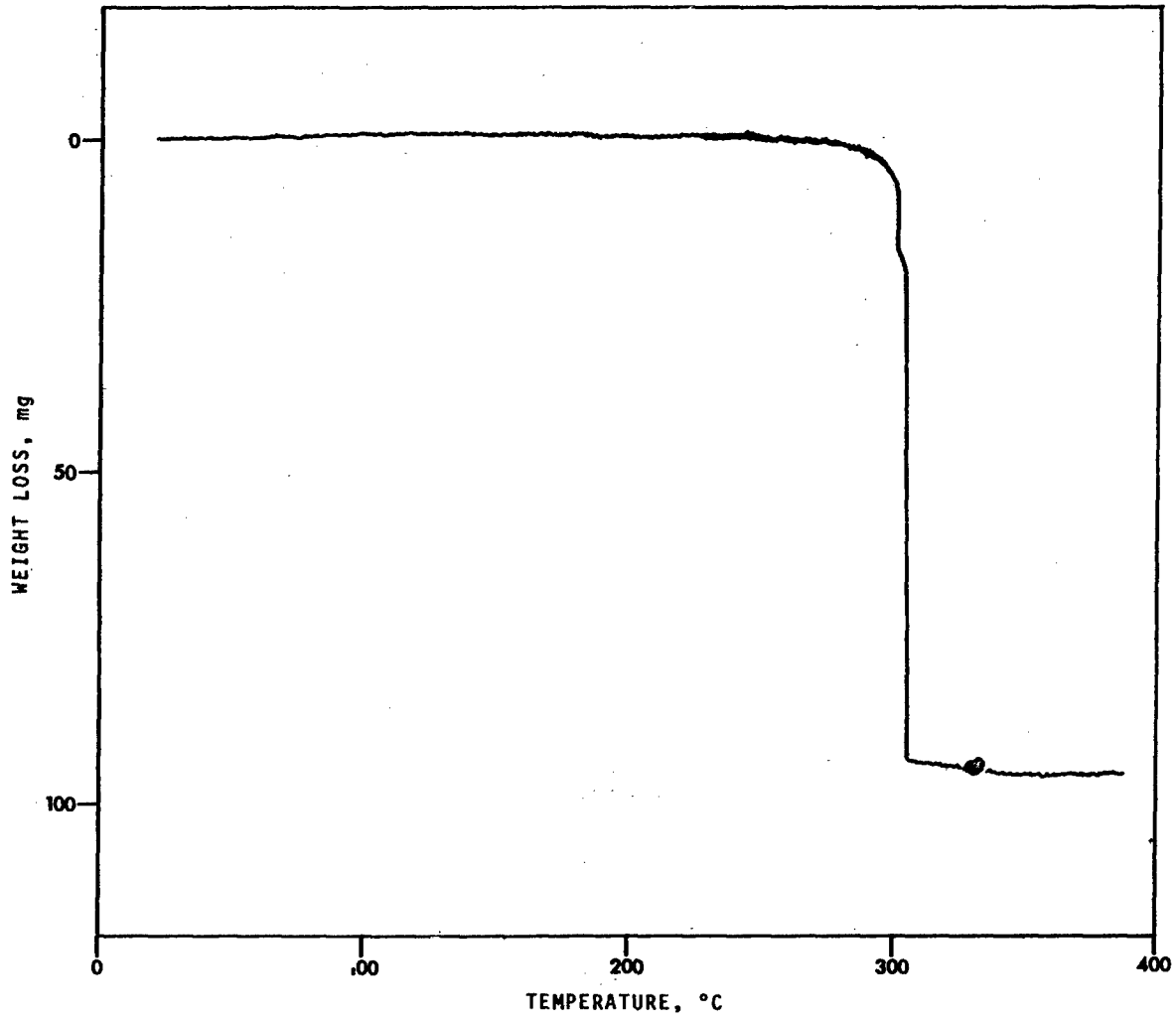

Figure 113. TGA Pattern of CNR AFE 110 Batch A014 (With Filler).

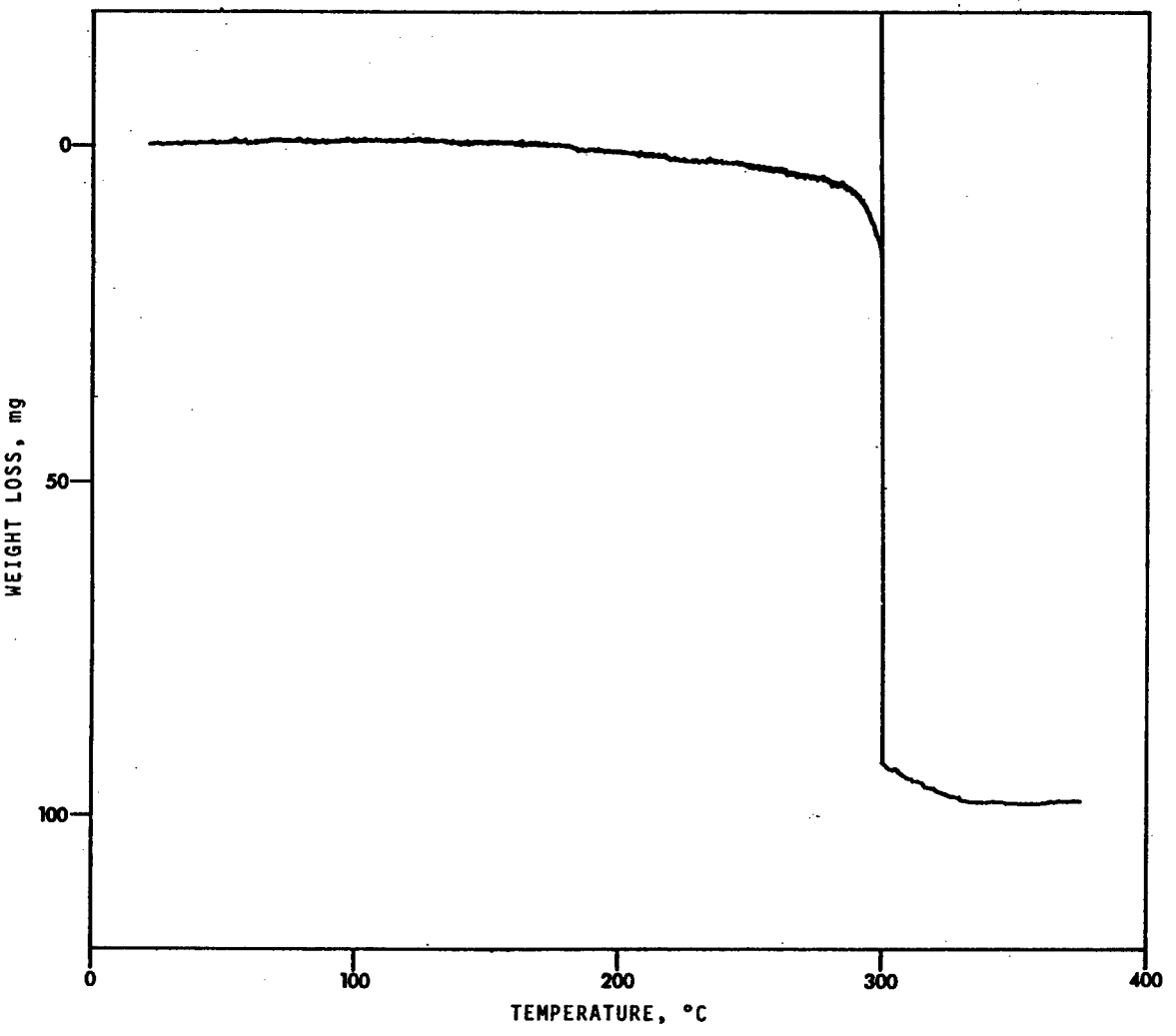

Figure 114. TGA Pattern of CNR Terpolymer Gum - A014F.

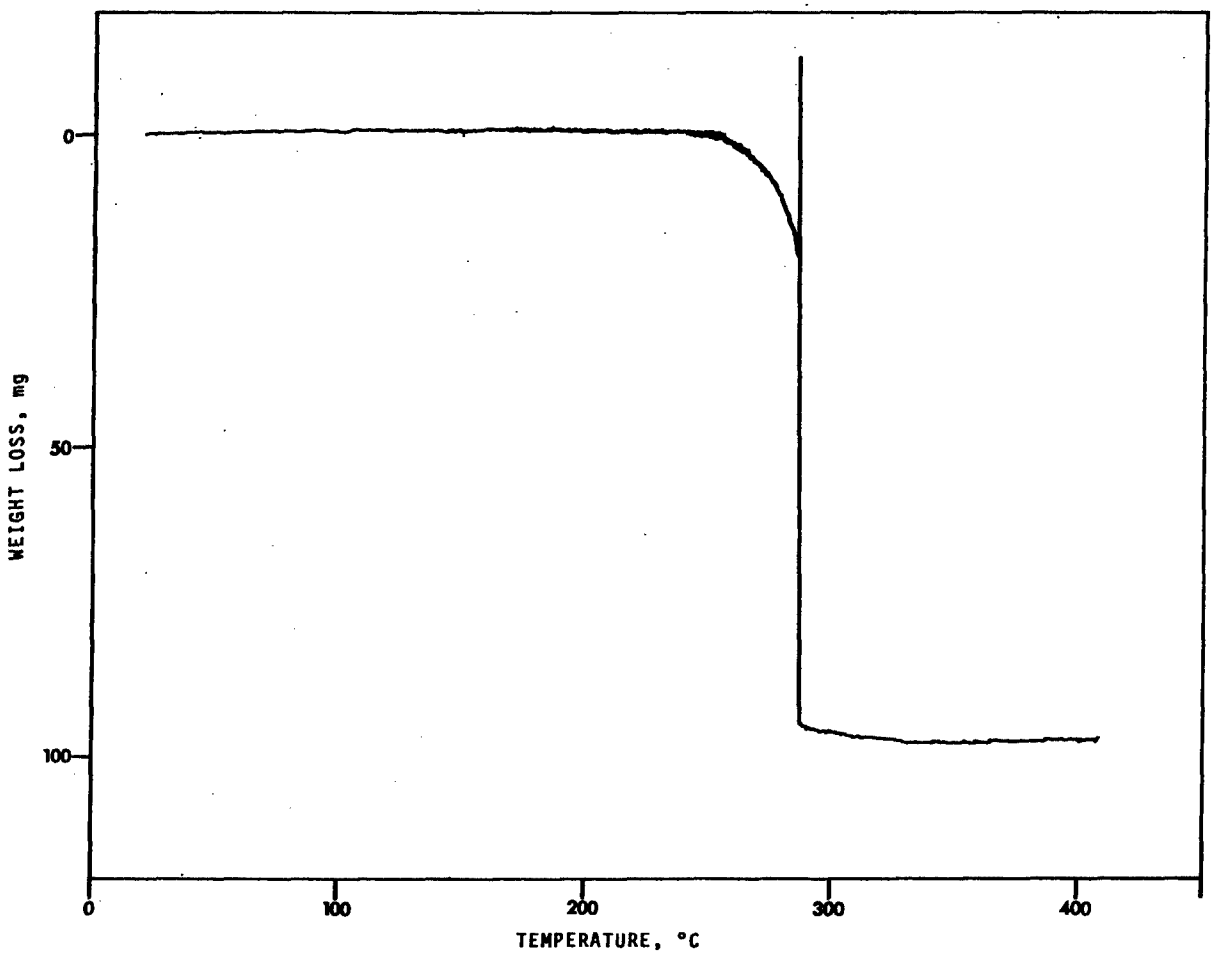

Figure 115. TGA Pattern of CNR Vulcanizate (Without Filler) 109-1164.

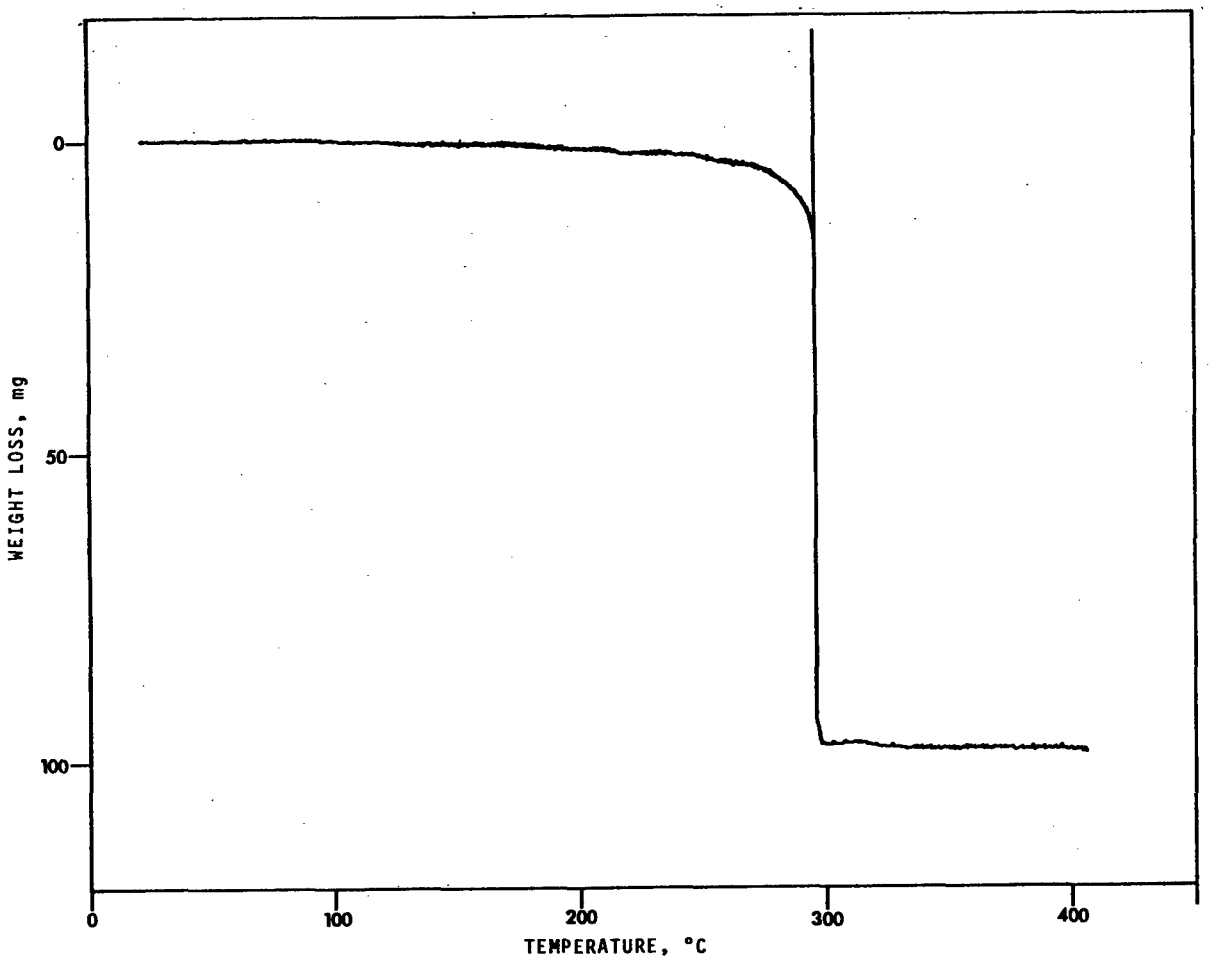

Figure 116. TGA Pattern of CNR GS #1 (MSC 1676).

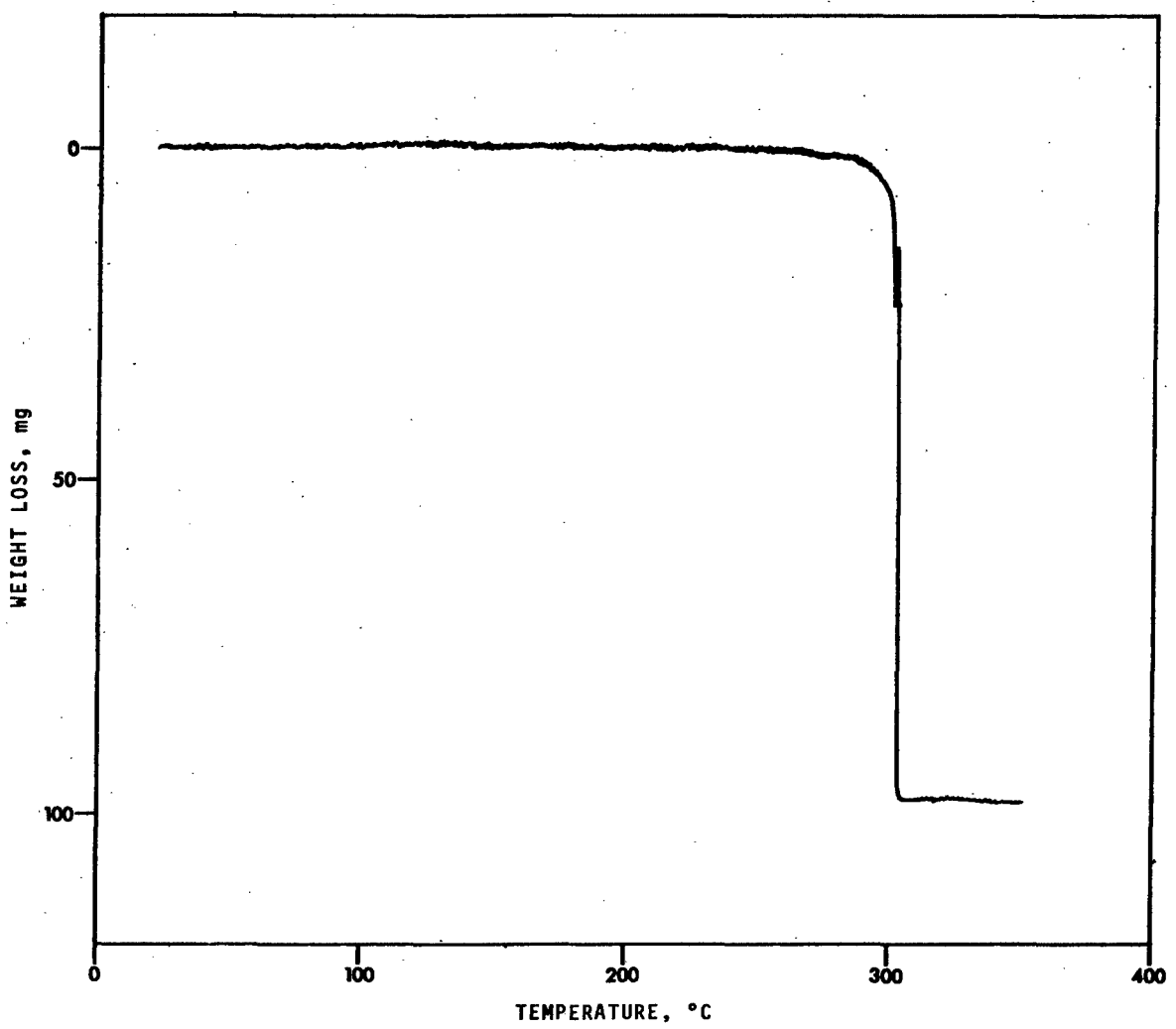

Figure 117. TGA Pattern of CNR AFE-110 - Batch A014 (MSC 1672).

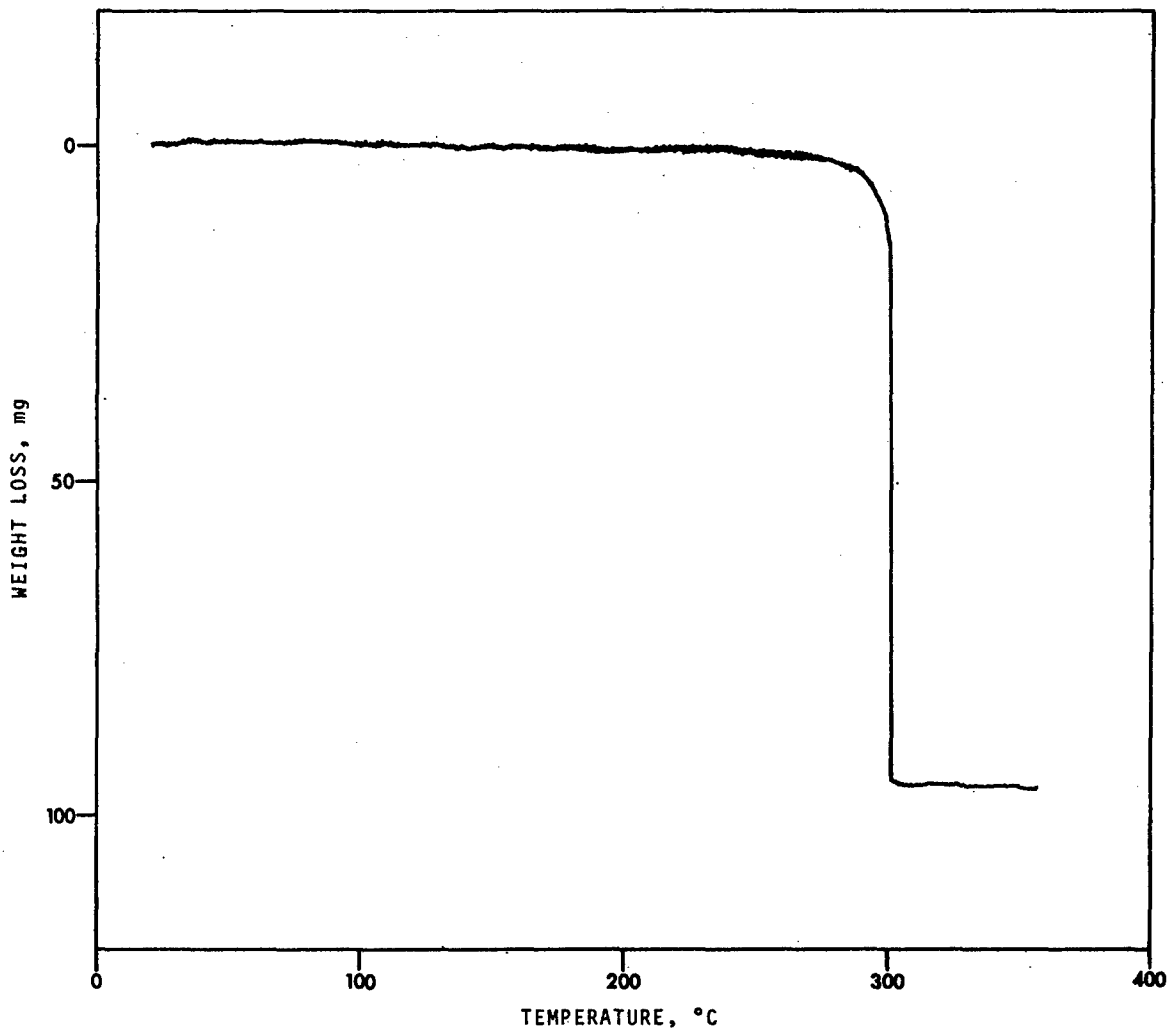

Figure 118. TGA Pattern of CNR AFE-110-A-006-1103 (MSC 1481).

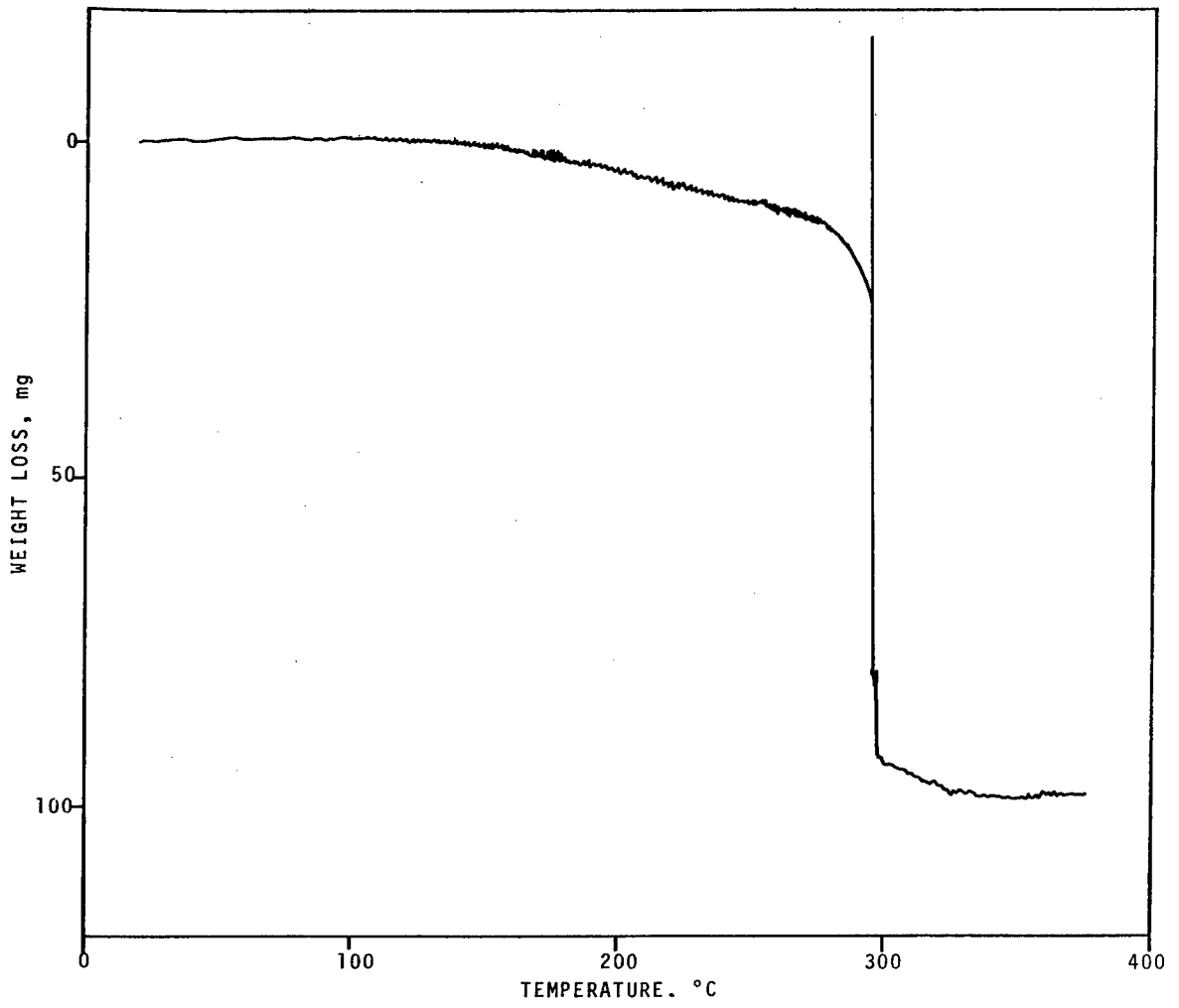

Figure 119. TGA Pattern of NRC-NA 3310-43-1 & 1B (MSC 1549).

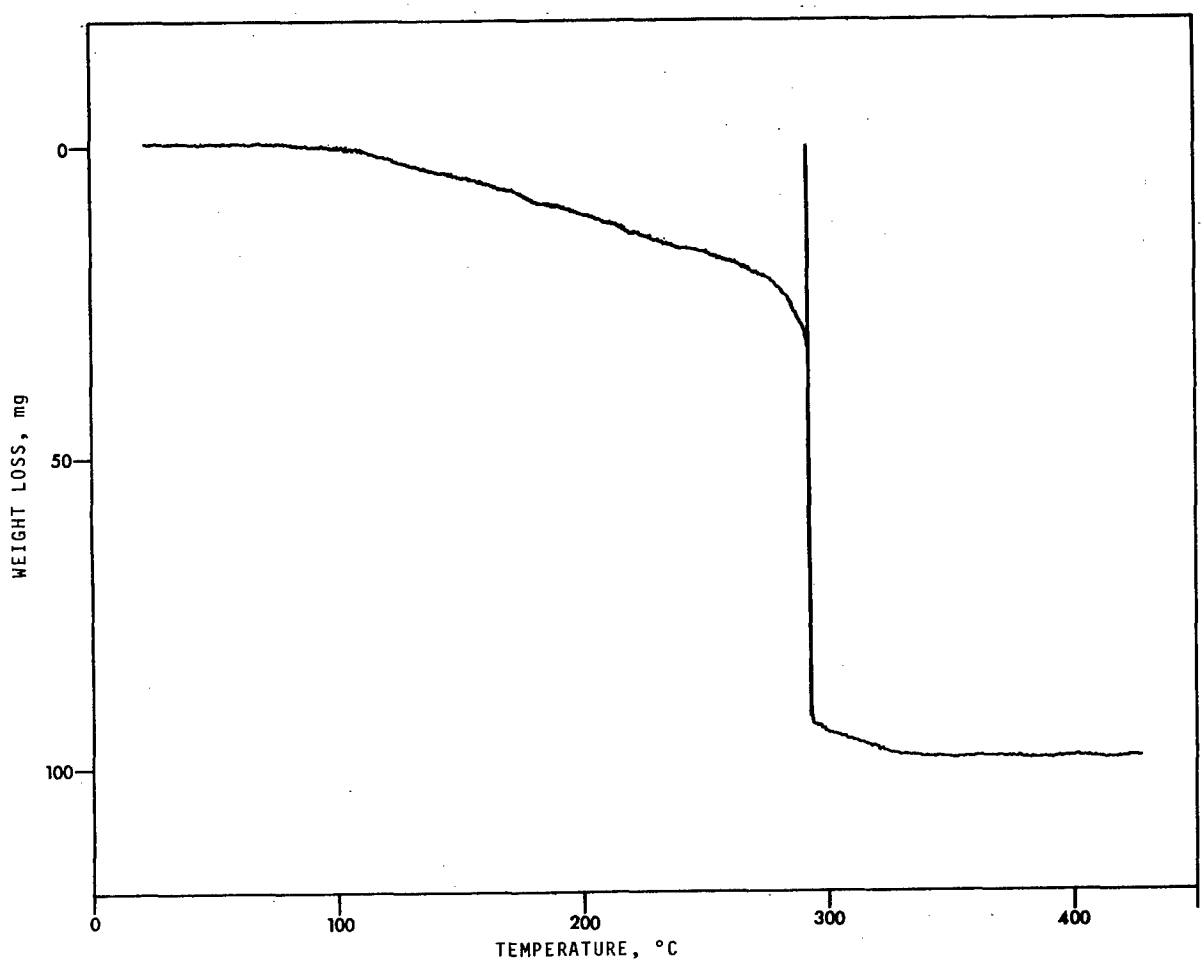

Figure 120. TGA Pattern of CNR Supplemental to MSC 1549.

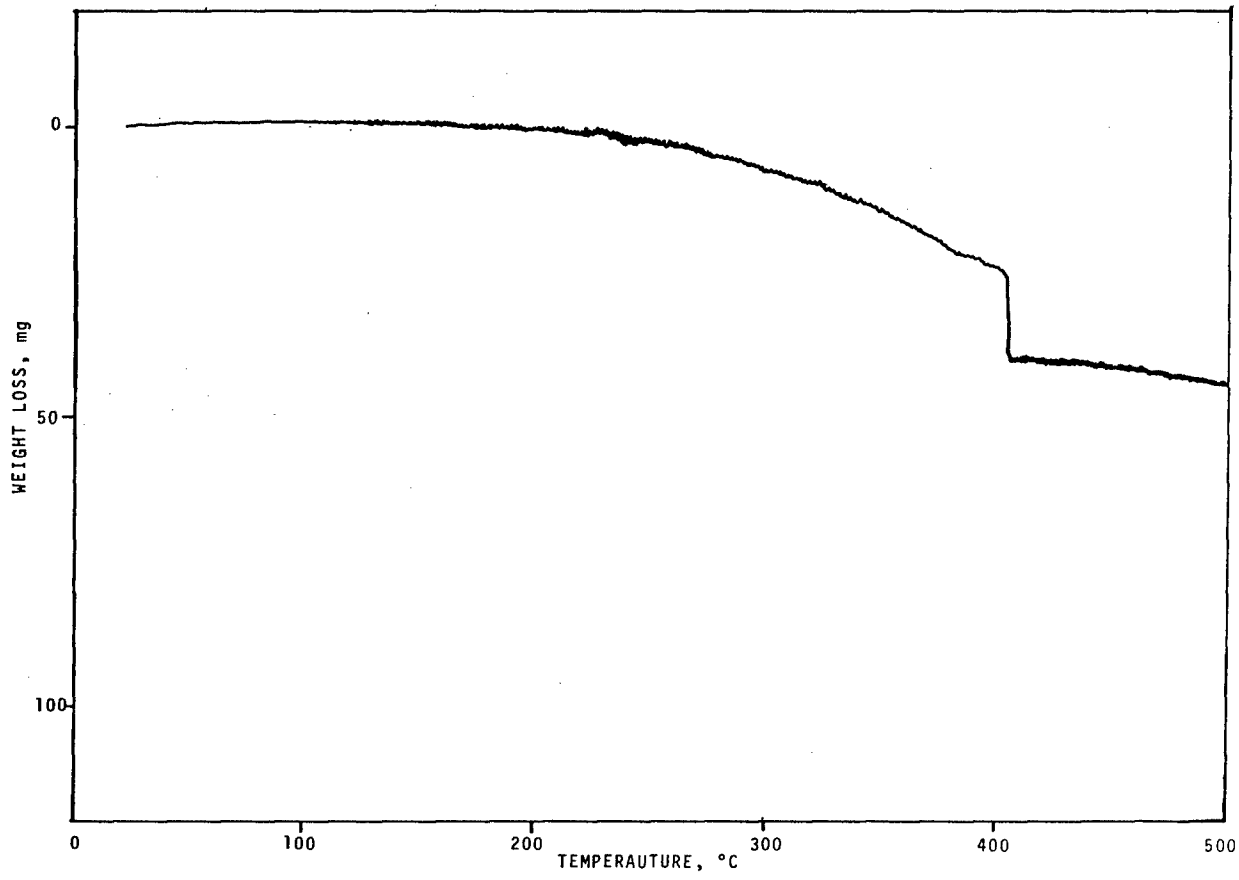

Figure 121. TGA Pattern of CNR (MSC 1475) (25-500°C).

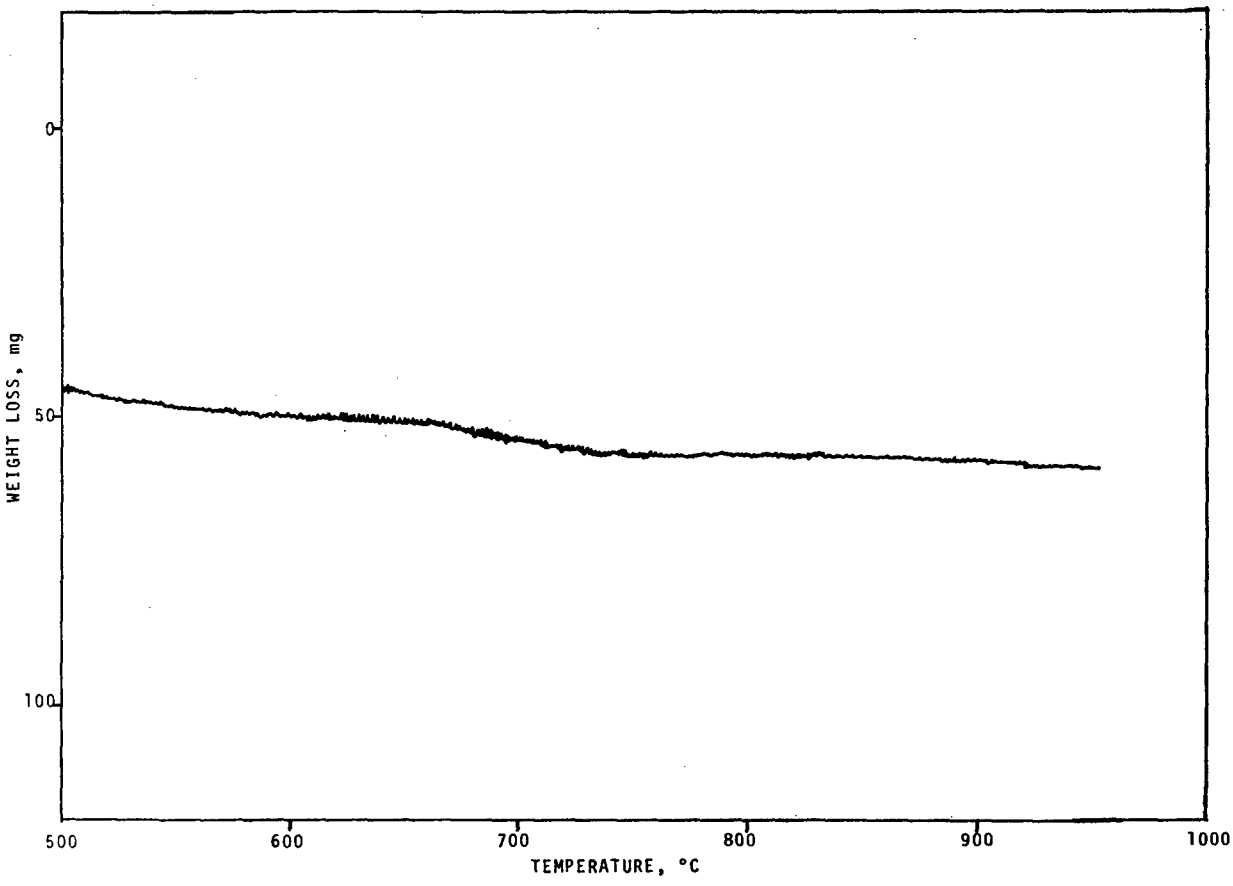

Figure 122. TGA Pattern of CNR (MSC 1475) (500-1000°C).

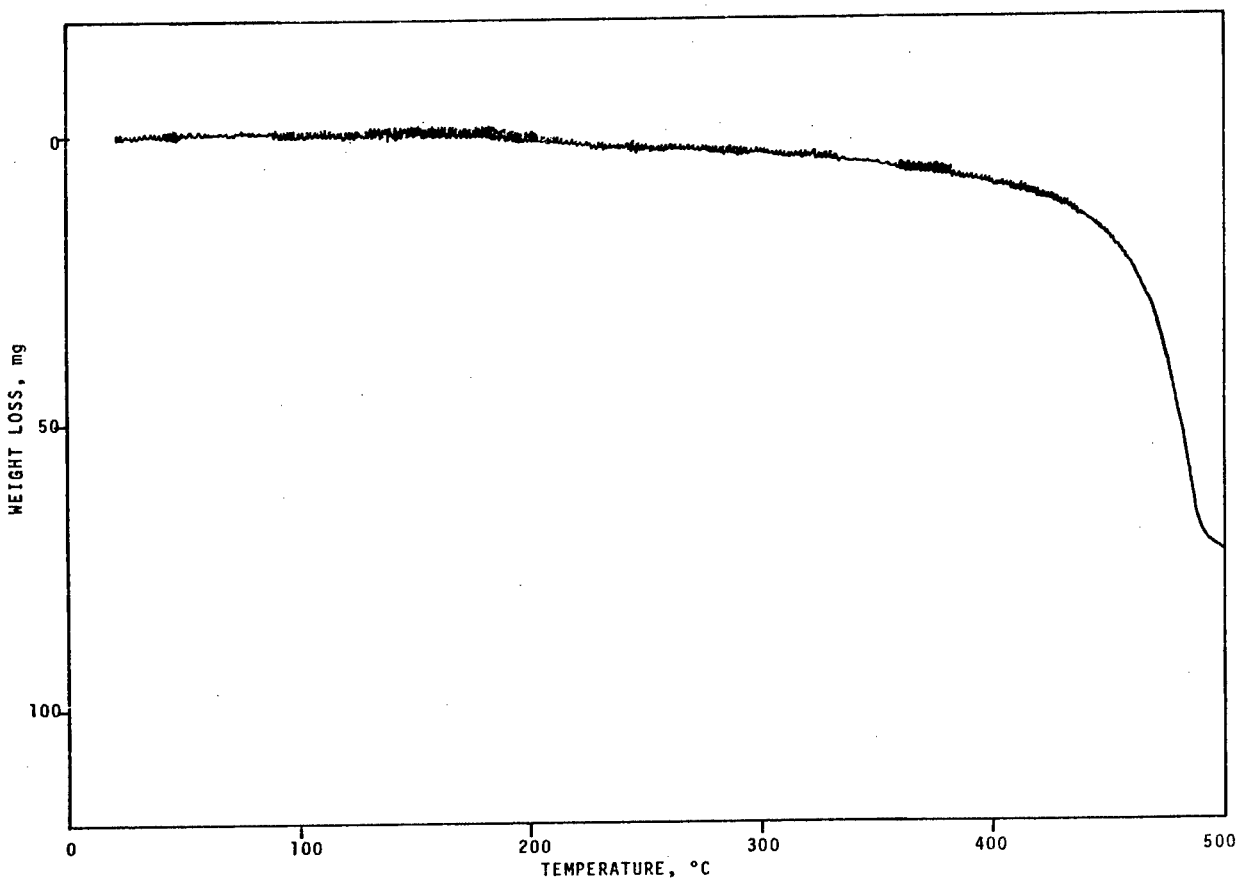

Figure 123. TGA Pattern of CNR (MSC 1480) (25-500°C).

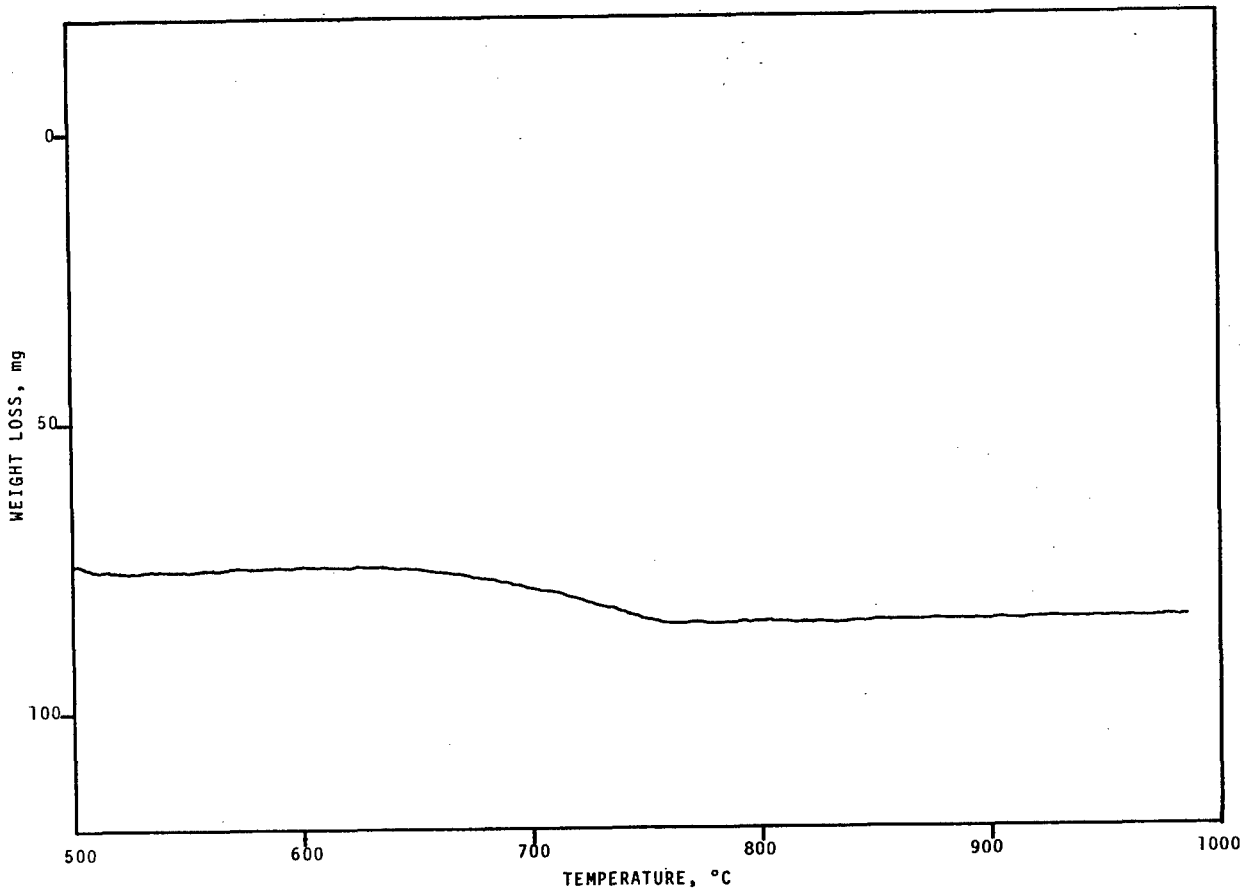

Figure 124. TGA Pattern of CNR (MSC 1480) (500-1000°C).

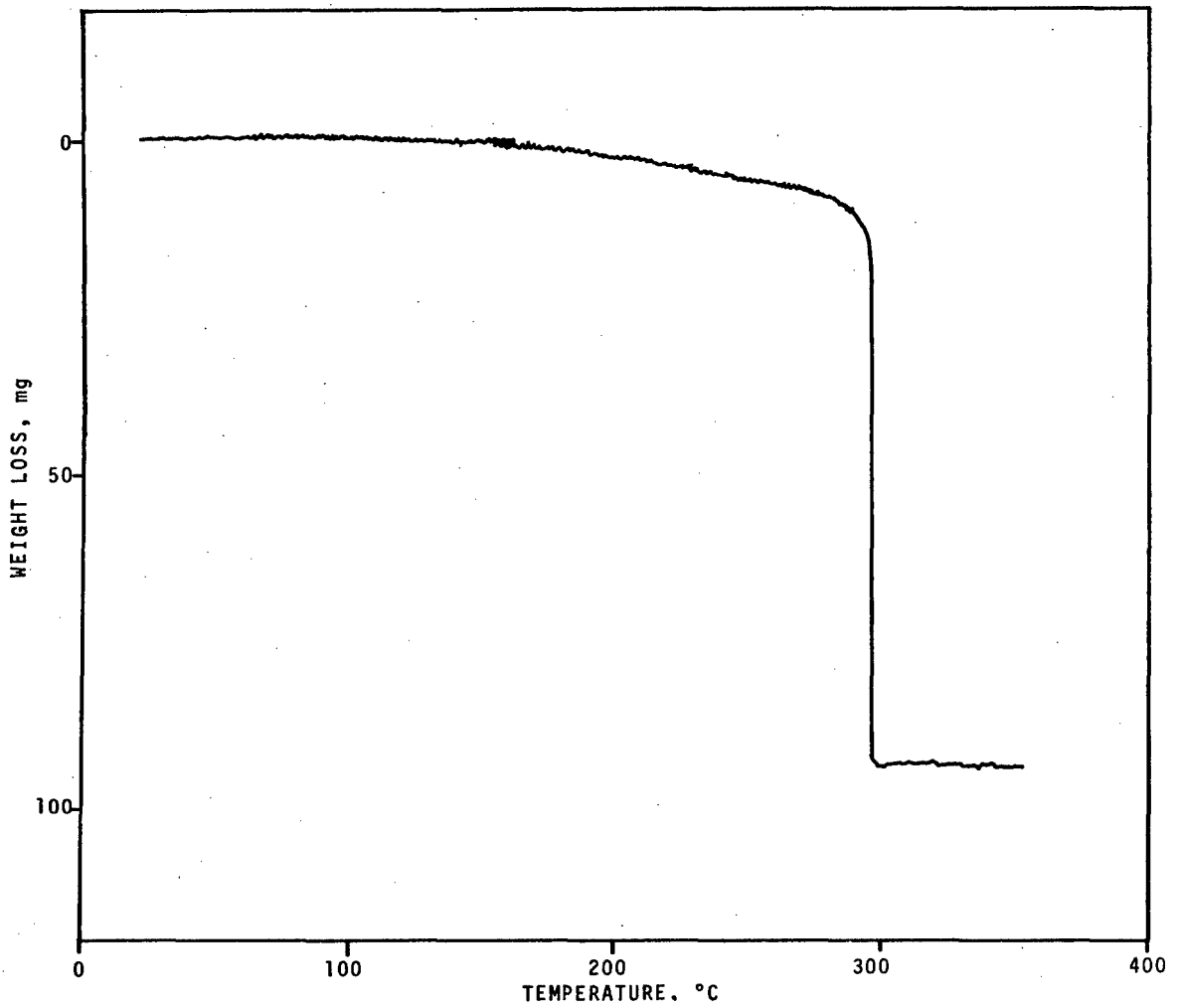

Figure 125. TGA Pattern of CNR Vulcanizate Cured at Room Temperature for 1 Hour, 200°F for 8 Hours (MSC 1673).

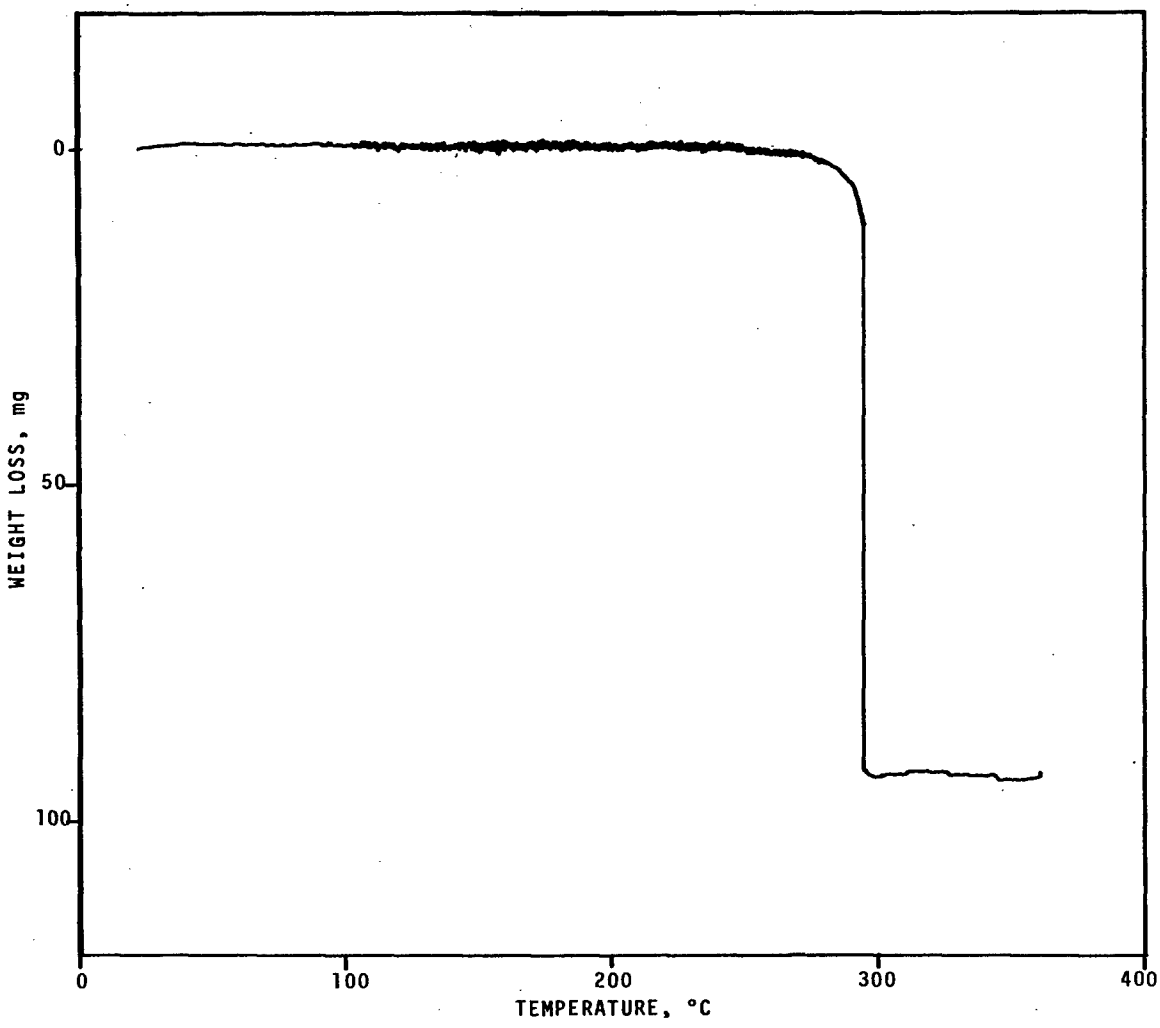

Figure 126. TGA Pattern of CNR Vulcanizate Cured at Room Temperature for 1 Hour, 300°F for 24 Hours, 350°F for 16 Hours (MSC 1674).

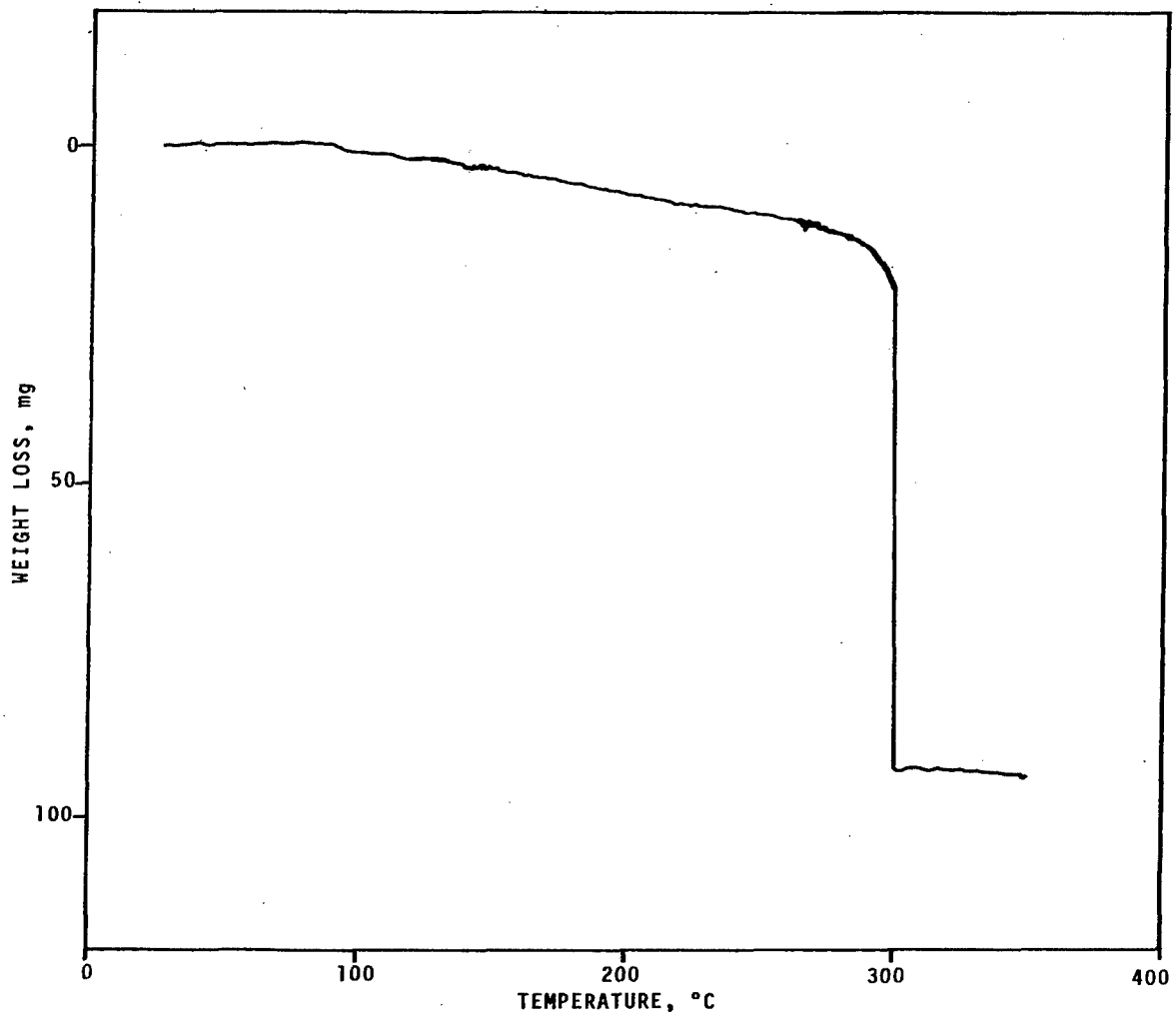

Figure 127. TGA Pattern of CNR Vulcanizate Cured at Room Temperature (MSC 1675).